

The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 73 Number 2

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THE SOCIETY OF DYERS AND COLOURISTS
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The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and Figures in Arabic numerals.

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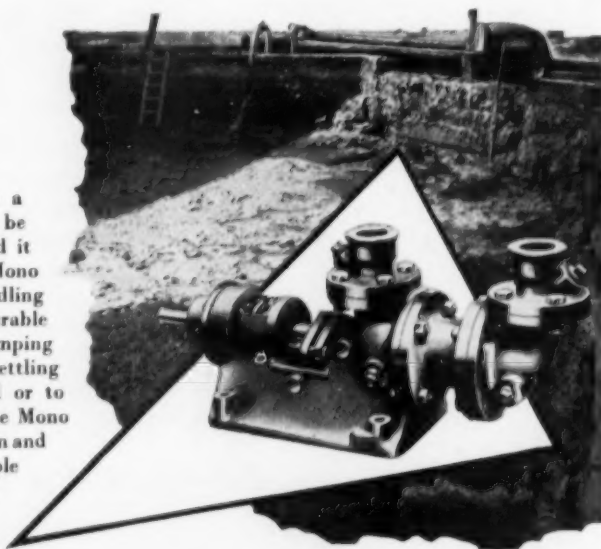
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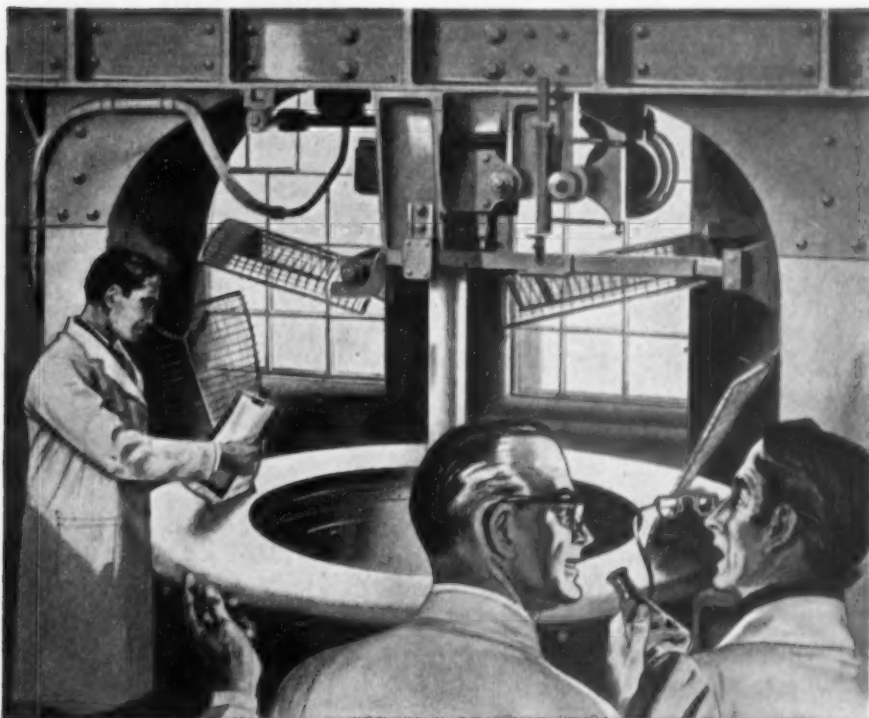
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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1-7 of the January 1957 and pages 313-317 of the July 1956 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138-9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

LECTURES

William Perkin, His Life and Work	<i>J. Boulton</i>
Dyeing of Man-made Fibres in the Carpet Trade	<i>R. C. Cheetham</i>
Application of Dyes to Acrylic Fibres	<i>B. Kramrisch</i>

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A Critical Appraisal of the International Daylight Fastness Test	<i>K. McLaren</i>
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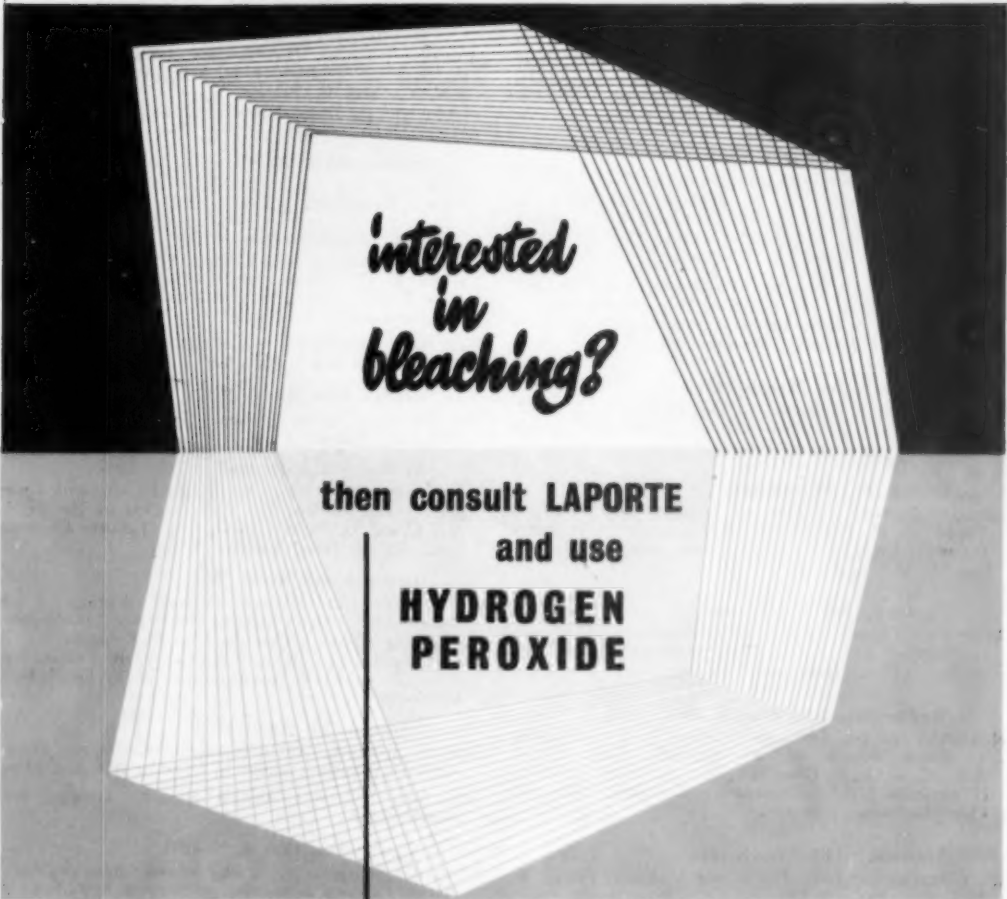
The Dyeing of Cellulose Acetate with Disperse Dyes — VI	<i>C. L. Bird and Miss P. Harris</i>
Measurement of Diffusion Coefficients of Azoic Coupling Components in Cellulose	<i>A. S. Dunn</i>
A Method of Isolation of the Cuticle Sheath from Wool Fibres	<i>R. L. Elliott and J. B. Roberts</i>
Light-fastness Assessments of Dyed Textiles and their Bearing upon the Mechanism of Fading	<i>C. H. Giles</i>
Alginate Hessian	<i>E. N. Woodward et al.</i>

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FORTHCOMING MEETINGS OF THE SOCIETY

Thursday, 28th February 1957

WEST RIDING SECTION. *The Applications of Fluid Beds to Dyeing.* J. Wilson, Esq., M.C., M.Sc. and F. G. Audas, Esq. (British Rayon Research Association). Victoria Hotel, Bridge Street, **Bradford**. 7.30 p.m.

Tuesday, 5th March 1957

LEEDS JUNIOR BRANCH. *Reactive Dyes. A New Approach to Cellulose Dyeing.* C. D. Weston, Esq. (Imperial Chemical Industries Ltd.). Departmental Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, **Leeds 2**. 3.30 p.m.

MANCHESTER JUNIOR BRANCH. *The Principles and Practice of Package Dyeing.* K. Limbert, Esq. (The Longclose Engineering Co. Ltd., Leeds). Manchester College of Science and Technology, **Manchester**. 7 p.m.

Thursday, 7th March 1957

BRADFORD JUNIOR BRANCH. *Flame Proofing.* J. R. W. Perfect, Esq. (Bradford Dyers' Association Ltd.). Technical College, **Bradford**. 7.15 p.m.

Friday, 8th March 1957

LONDON SECTION. *Soiling of Natural and Synthetic Fibres.* G. G. Taylor, Esq., B.Sc., A.Inst.P. (Clayton Dyestuffs Co. Ltd.). Royal Society, Burlington House, **London W.1**. 6 p.m.

Tuesday, 12th March 1957

SCOTTISH SECTION. *Recent Developments in Wool Dyeing.* G. H. Lister, Esq., Ph.D., B.Sc. (Sandoz Products Ltd.). St. Enoch Hotel, **Glasgow**. 7.15 p.m.

Wednesday, 13th March 1957

NORTHERN IRELAND SECTION. *Problems in Wet Processing of Rayon Blends.* H. D. Edwards, Esq., Ph.D., A.R.I.C. and R. C. Cheetham, Esq., A.M.C.T., F.R.I.C. (Courtaulds Ltd.). Thompson's Restaurant, Donegall Place, **Belfast**. 7.30 p.m.

Thursday, 14th March 1957

WEST RIDING SECTION. *The Dyeing of Acrylic Fibres.* K. Meldrum, Esq., B.Sc., A.R.I.C. (Courtaulds Ltd.). Victoria Hotel, Bridge Street, **Bradford**. 7.30 p.m.

Saturday, 16th March 1957

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Woodbourne House Hotel, **Belfast**. Details later.

Tuesday, 19th March 1957

HUDDERSFIELD SECTION. Annual General Meeting. Silvios Cafe, Westgate, **Huddersfield**. 7.30 p.m.

Wednesday, 20th March 1957

MIDLANDS SECTION. *Developments in Sizing as they affect the Dyer and Finisher.* J. H. MacGregor, Esq., Ph.D., F.R.I.C., and E. France, Esq., A.R.C.S., A.M.C.T. (Joint with the British Association of Chemists.) Midland Hotel, **Derby**. 7 p.m.

Friday, 22nd March 1957

BRADFORD JUNIOR BRANCH. Annual Dance. Connaught Rooms, **Bradford**.

Thursday, 28th March 1957

MIDLANDS SECTION. *The Soiling of Natural and Synthetic Fibres.* G. G. Taylor, Esq., B.Sc., A.Inst.P. (Joint with the Textile Institute.) Carpet Trades Canteen, **Kidderminster**. 7.30 p.m.

WEST RIDING SECTION. Annual General Meeting. Victoria Hotel, Bridge Street, **Bradford**. 7.30 p.m.

Friday, 29th March 1957

The Society's Annual Dinner. The Grosvenor House, Park Lane, **London**.

Friday, 5th April 1957

LONDON SECTION. *Some Aspects of Bleaching with Hydrogen Peroxide and Peracetic Acid.* L. Chesner, Esq., B.Sc., A.T.I. and G. C. Woodford, Esq. (Laporte Chemicals Ltd.). George Hotel, **Luton**. 7 p.m.

Tuesday, 9th April 1957

SCOTTISH SECTION. Annual General Meeting. 7 p.m. Followed by ordinary Meeting at 7.30 p.m. *Developments in Sizing as they Affect the Dyer and Finisher.* J. H. MacGregor, Esq., Ph.D., F.R.I.C. and E. France, Esq., A.R.C.S., A.M.C.T. (Courtaulds Ltd.). St. Enoch Hotel, **Glasgow**.

Friday, 12th April 1957

LONDON SECTION. Annual General Meeting and Dinner. Aldwych Brasserie, Aldwych, **London W.C.2**. 6.30 p.m.

MIDLANDS SECTION. Annual Dinner. George Hotel, **Nottingham**. 7 p.m.

Wednesday, 17th April 1957

MIDLANDS SECTION. . . . and inwardly digest (a Dissertation on Technical Literature). S. Burgess, Esq., F.S.D.C., A.M.C.T. (Preceded at 6.30 p.m. by the Annual General Meeting of the Section). King's Head Hotel, **Loughborough**. 7 p.m.

Saturday, 20th April 1957

BRADFORD JUNIOR BRANCH. Annual General Meeting. Technical College, **Bradford**. 10.15 a.m.

Friday, 3rd May 1957

MIDLANDS SECTION. The Mercer Lecture of the Society. Grand Hotel, **Leicester**. 7 p.m.

JOURNALS WANTED

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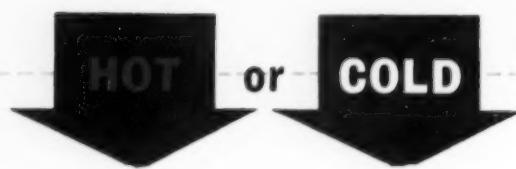
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OF THE

Society of Dyers and Colourists

Volume 73 Number 2

FEBRUARY 1957

Issued Monthly

COMMUNICATIONS

The Chemistry of Esters of Leuco Vat Dyes

II—A Comparison of the Rates of Oxidation with the Standard Redox Potentials of the Parent Vat Dyes

S. AINSWORTH and A. JOHNSON

The rates of oxidation, with acidic hydrogen peroxide, of a number of disulphuric esters of leuco vat dyes have been measured and compared with the standard redox potentials of the parent vat dye-leuco vat dye systems. On the basis of this comparison the esters may be divided into two classes according to the relative importance of oxidation or hydrolysis in the development sequence.

INTRODUCTION

In Part I¹ it was shown that the oxidation of sulphuric esters of leuco vat dyes* under acidic conditions occurs more rapidly than the removal of sulphate groups, presumably by hydrolysis, in acid alone. Nevertheless, it was shown that with hydrogen peroxide, which can act as an oxidising agent in both acidic and alkaline solutions, oxidation occurred only in acid. It was therefore concluded that hydrolysis of part of the ester was essential before oxidation of the remainder could commence, the products of hydrolysis acting as oxidation catalysts.

The overall rate of oxidation of a vat ester with acidic hydrogen peroxide is thus influenced by two factors, viz. hydrolysis and oxidation. The present study is an attempt to determine the relative importance of these two factors.

The ease with which sulphate groups may be removed from a vat ester molecule will be influenced by structural features of the rest of the molecule insofar as these affect the electron density at the sulphate group. It can be shown that the effects of structure on the two reactions in question will be opposed, electron accession to the reacting site favouring oxidation and electron recession favouring hydrolysis. It was thought, therefore, that a comparison between the relative rates of oxidation, with acidic hydrogen peroxide, of a series of vat esters and the corresponding electron densities at the sulphate groups would indicate the relative importance of hydrolysis and oxidation in the overall development sequence. In order to do this it is necessary to choose a suitable parameter by which to measure the electron density at the

reaction site. For the reasons given below it was decided that the standard redox potentials of the parent vat dye-leuco vat dye systems would be adequate for this purpose.

Effect of Structure on Hydrolysis

The rates of hydrolysis of a selected series of leuco vat dye esters have not been measured. However, those of a series of substituted phenyl potassium sulphates (which differ from vat esters in that they contain only one ester group per molecule) have been measured by Burkhardt *et al.*², who found that the velocity constants decreased in the following order of substituents: $p\text{-NO}_2 > o\text{-NO}_2 > m\text{-NO}_2 > m\text{-Cl} > o\text{-OCH}_3 > o\text{-Cl} > p\text{-Br} > p\text{-Cl} > m\text{-OCH}_3 > \text{-H} > m\text{-CH}_3 > o\text{-CH}_3 > p\text{-CH}_3 > p\text{-OCH}_3$; i.e. hydrolysis is easier with electron-attracting substituents. Burkhardt also found a linear relation between $\log k$ and $\log K$ for the corresponding benzoic acids (k = velocity constant for hydrolysis, K = acid dissociation constant); that is to say, there is a linear relation between the logarithm of the velocity constant for hydrolysis and the appropriate Hammett substitution constant σ . Hydrolysis becomes more rapid as the value of σ becomes more positive (Fig. 1).

Effect of Structure on Oxidation

Since oxidation is essentially the removal of electrons from the substance oxidised (irrespective of the removal or addition of other atoms or groups of atoms), it follows that, within a properly selected series of compounds, oxidation will become easier as the electron density at the site of oxidation increases. If this principle is applied to the oxidation of a side-chain directly attached to an

* Subsequently referred to as vat esters as in Part I.

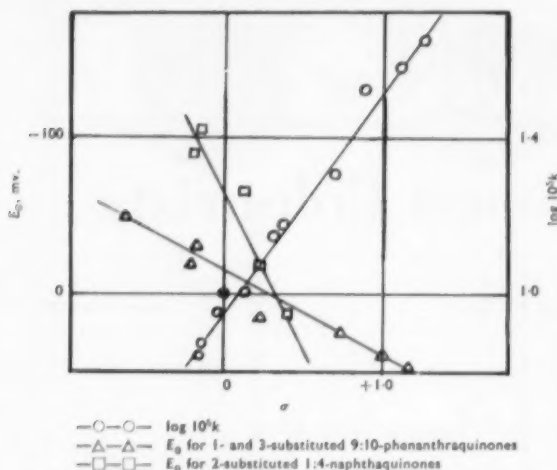


Fig. 1.—Relation between Hammett Constants (σ), Rate Constants (k) for Hydrolysis of Substituted Potassium Aryl Sulphates, and Redox Potentials (E_0) for Substituted Quinones

aromatic nucleus, then oxidation should occur more readily as other substituents are made more positive, i.e. as the value of σ becomes more negative. A direct numerical verification of this principle is, however, not readily obtainable, since oxidation of an aromatic compound often involves transition to a quinonoid form. Oxidation has then occurred at two sites, and the substituent cannot be in the same position relative to both.

However, even with quinol-quinone systems a general agreement with the anticipated effects of substituents has been observed. Thus, the Fiesers³ have shown that, with minor exceptions, the stability of a quinone relative to the quinol is favoured by positive substituents. Moreover, Carter, in a private communication to Evans⁴, has pointed out a linear relation between the standard redox potentials of substituted quinones and the appropriate Hammett constants. This is shown in Fig. 1. Although Evans⁴ was unable to find any theoretical justification for Carter's results, it is sufficient for the present work that there is a demonstrable relationship between the redox potentials of a series of substituted quinones and the relevant Hammett constants.

If the relation observed by Carter is valid, it follows that, for a suitable quinone-quinol system, the standard redox potential E_0 may be used in place of σ as a measure of the effect of substituents on the electron density at the two oxygen atoms. Furthermore, E_0 may be used in more complex structures where the appropriate value for σ cannot be evaluated.

The use of redox potential as a measure of structural influences in attempts to relate the structure and the reactivity of quinones and quinols has been employed by other workers. Thus Bolland and ten Have⁵ have observed a linear relation between redox potential and inhibitor efficiency for a series of quinols used to inhibit the oxidation of ethyl linoleate. Waters and Dunn⁶

have shown that inhibition of the autoxidation of benzaldehyde by quinones may be correlated with their redox potentials.

Structural Influences in Vat Esters

In a vat ester both hydroxyl groups of the quinol are esterified, the sulphate groups being removed irreversibly by either oxidation or hydrolysis. Thus, in comparing the rates of these reactions with the redox potentials of quinone-quinol systems we are comparing the effect of substituents on rate constants on the one hand with their effect on an equilibrium constant on the other. It is well known that such comparisons are valid, so that it is reasonable to assume that the reactivity of a series of vat esters towards oxidation will vary in the same sense as the appropriate values of E_0 . For the reasons given, the effect of structure on the rates of hydrolysis will be in the opposite sense (see Fig. 1).

Ideally, this study should be made on a series of esters having the same nucleus and differing only in the nature and the position of substituents. Since the time available for the present work was insufficient for the preparation of such a series, a number of purified commercial vat esters in the indigo and thioindigo series was used. In this case, therefore, a comparison of reactivity in terms of redox potential involves the assumption, made above, that E_0 may be used in complex structures where the appropriate value of σ cannot be evaluated.

EXPERIMENTAL

Materials

VAT ESTERS—Experiments were performed with the vat esters listed in Table I. The thioindigo derivative (No. 5) was prepared by the method described in Part I¹; the remainder were commercial samples. All the dyes were purified by first washing with chloroform to remove free vat dye, and then extracting with acetone. The vat ester was precipitated from the acetone extract by careful addition of ether or chloroform. This was repeated three times, the final product being washed with ether and dried. Light was excluded as much as possible.

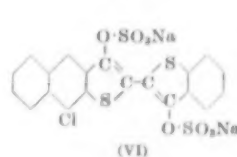
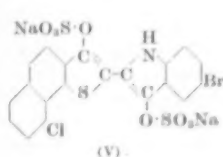
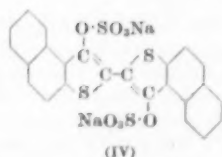
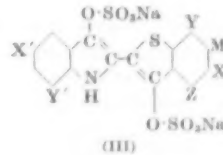
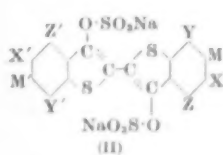
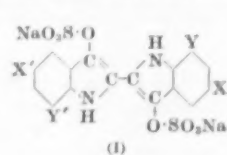
VAT DYES—These were prepared from the corresponding purified esters by careful oxidation with nitrous acid, followed by thorough washing with water and finally acetone and ether.

PYRIDINE—Freshly distilled pyridine was refluxed for several hours, first over anhydrous ferric chloride to destroy oxidisable impurities and subsequently over sodium hydroxide pellets. It was then distilled through an efficient column, the fraction boiling at 118°C. being collected and stored over potassium hydroxide pellets.

PYRIDINE HYDROCHLORIDE—Dry hydrogen chloride was passed into anhydrous pyridine and the concentration of the solution thus obtained determined by titration against sodium hydroxide to the phenolphthalein end-point. For use, this solution was diluted to 0.2 M. by adding dry pyridine.

TABLE I
 Vat Esters used for Rate-of-reaction Measurements

No.	Name	Structure
1	Soledon Indigo LLS (ICI)	I: $X = X' = Y = Y' = H$
2	Indigosol OR (DH)	I: $X = Br; X' = Y = Y' = H$
3	Indigosol O4B (DH)	I: $X = X' = Y = Y' = Br$
4	Soledon Orange RS (ICI)	II: $M = M' = O-C_2H_5; X = X' = Y = Y' = Z = Z' = H$
5	Vat ester of thioindigo (laboratory preparation)	II: $M = M' = X = X' = Y = Y' = Z = Z' = H$
6	Soledon Red 3BS (ICI)	II: $X = X' = Cl; Y = Y' = CH_3; M = M' = Z = Z' = H$
7	Indigosol Brilliant Pink I3B (DH)	II: $M' = X = Y = Cl; Z = Z' = CH_3; M = X' = Y' = H$
8	Soledon Pink FFS (ICI)	II: $M = M' = Cl; Z = Z' = CH_3; X = X' = Y = Y' = H$
9	Indigosol Scarlet IB (DH)	II: $M = O-CH_3; M' = Cl; Z' = CH_3; X = X' = Y = Y' = Z = H$
10	Indigosol Printing Violet IRR (DH)	III: $M = X' = Y' = Cl; Z = CH_3; X = Y = H$
11	Indigosol Printing Violet IBBF (DH)	III: $M = X = X' = Y = Y' = Cl; Z = H$
12	Cibantone Brown GS (Ciba)	IV
13	Soledon Grey BS (ICI)	V
14	Indigosol Printing Purple IR (DH)	VI



Measurement of Rate of Reaction

The colorimetric methods used earlier (Part I¹) were unsuitable because of the continuous precipitation of vat dye. However, the development of a vat ester results in the liberation of bisulfate ions, with an equivalent increase in acidity of the solution. Thus, with suitable concentrations of ester and acid it is possible to follow the extent of the reaction by titrating the liberated bisulfate against standard alkali (see Burkhardt *et al.*²).

This procedure necessitated the use of considerably higher concentrations of vat ester and hydrogen peroxide than were used before. For this reason air dissolved in the reaction solution would have a relatively small effect, and so it was not removed.

The experiments were performed in a bifurcated vessel (Fig. 2) closed with a rubber bung. Hydrochloric acid (50 ml., usually 0.1 N.) was placed in limb A, the vessel placed in a thermostat at 35°C., and, after 30 min., 50 ml. of a solution of the vat ester (0.05 N.) in hydrogen peroxide (usually 0.246 N.) pipetted into B. After 5 min. the solutions were mixed by inverting the vessel and shaking. From time to time 5-ml. portions were withdrawn and either titrated directly against 0.012 N. sodium hydroxide solution (direct titration method) or run into 5 ml. of 0.11 N. sodium hydroxide solution to stop the reaction and

subsequently titrated against 0.01 N. hydrochloric acid (back-titration method). Light was excluded throughout the reaction.

No more than 5 min. could be allowed for temperature stabilisation of the vat ester solution for fear of premature decomposition. However, the temperature of the final reaction solution did

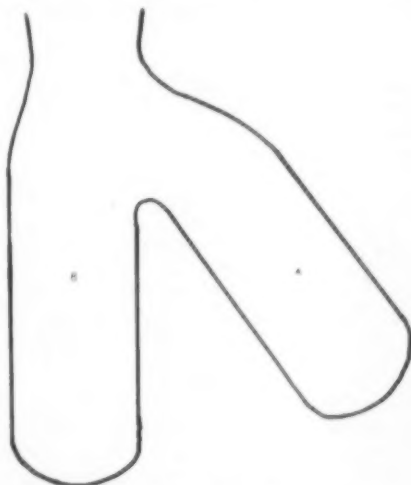


FIG. 2—Reaction Vessel

not differ from that of the bath by more than 1°C.

Bromothymol Blue, for red and orange, and Methyl Red, for blue vat dyes, were used as indicators, since they gave the sharpest colour change in the presence of suspended vat dye. After some experience, the end-point could be judged with certainty and reproducible results obtained. The majority of the experiments were duplicated.

Measurements of Redox Potentials

The redox potentials of the vat dye-leuco vat dye systems were measured in anhydrous pyridine by the method due to Gupta⁷ and also used by Marshall and Peters⁸. The apparatus was the same as that used by the latter authors, the leuco forms of the dyes being titrated against pyridine solutions of anhydrous ferric chloride. Concentrations were adjusted to suit the solubility of the particular dye and to give a final titre of ca. 2 ml. The concentration of ferric chloride did not exceed 1.5 g./litre.

RESULTS AND DISCUSSION

Effects of Hydrochloric Acid and Hydrogen Peroxide

In a series of experiments with Soledon Orange RS (0.025 N.) the concentrations of hydrochloric acid and hydrogen peroxide were varied within the range 0.0125–0.0775 N. for the acid and 0.03–0.123 N. for the peroxide. Increasing the concentration of either increased the rate of reaction, but at acid concentrations above 0.05 N. the rate curves tended to be irregular. From these results the values 0.05 N. for the concentration of hydrochloric acid, 0.123 N. for hydrogen peroxide, and 0.025 N. for the vat ester were selected as standard conditions for subsequent experiments.

In Part I it was reported that the vat ester from indigotetrasulphonic acid was oxidised more rapidly by hydrogen peroxide which had been stored in brown glass bottles than by that which had been stored in clear glass bottles. This comparison was repeated with Soledons Orange RS, Red 3BS, and Pink FFS, selected at random. The results (determined by back-titration) are shown in

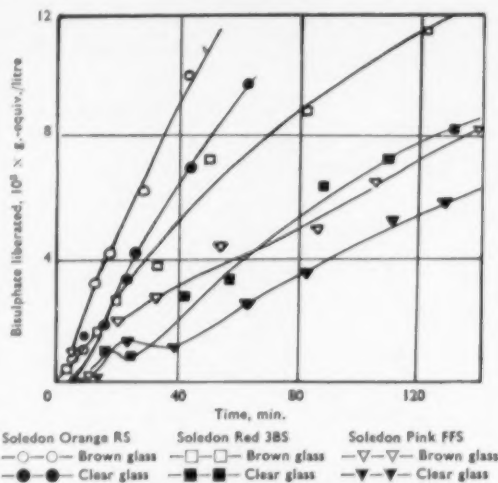


FIG. 3—Comparison of Oxidations with Hydrogen Peroxide from Brown and Clear Glass Bottles

Fig. 3 and clearly confirm the effect. Not only is the rate increased but the induction period is diminished considerably and the irregularities which are characteristic of the back-titration method (see later) are almost eliminated.

Comparative Rates of Oxidation

Rate-of-oxidation curves for the vat esters listed in Table I, when measured by the direct titration method, had the general shape illustrated in Fig. 4. After a short induction period the slope of the curve increases more or less rapidly to a point of inflection, following which the slope decreases to the end of the reaction. With some members, chiefly from Class B (below), which in general reacted slowly, the curve was not followed beyond the point of inflection. It was found that the vat esters could be divided into two classes, those in which the inflection occurred at an early stage in the reaction (Class A) and those in which the inflection was much later (Class B). This classification is shown in detail in Table II.

TABLE II
Classification of Vat Esters

Dye No. (Table I)	Class	Data from Standard Oxidations		Redox Potential, $-E_0$ (mv.)	
		SO_4^{2-} (%) [*]	$t_{20\%}$ †	Present Work	Marshall and Peters ⁸
4	A	3.5	37	253	210
1	A	3.5	50	217	220
10	A	4	190	156	—
5	A	4	250	148	160
6	A	4	80	107	110
11	A	7.5	220	80	—
12	A	7.5	18	174	220
2	A	8	30	166	—
3	B	22	63	68	70
7	B	24	92	189	—
9	B	24	210	230	—
8	B	36	130	215	190
13	B	48	60	128	—
14	B	55	100	157	—

* SO_4^{2-} (%) = Molar % of combined sulphate liberated at point of inflection.

† $t_{20\%}$ = Time (min.) for liberation of 20% of combined sulphate.

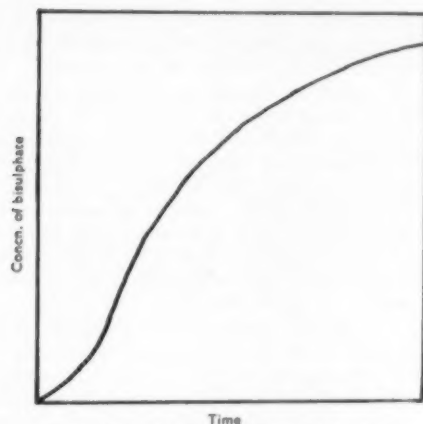


FIG. 4—General Shape of Rate-of-oxidation Curve

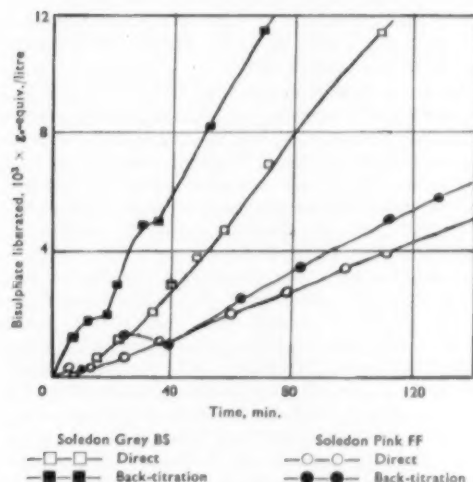


FIG. 5—Comparison of Direct and Back-titration Methods

The results obtained by the direct titration method were reproducible and gave rise to smooth rate-of-reaction curves. When the rates were measured by the back-titration method, however, the rate curves showed pronounced irregularities (Fig. 5). Although the reproducibility was not so good by this method, it was nevertheless established that the rate curves obtained by back titration always lay above those obtained directly (Fig. 5). This confirms the observation made with the vat ester from anthraquinone-2-sulphonic acid¹ that an intermediate product obtained by acidic oxidation can react further in alkaline solution.

The relative rates of reaction under the standard conditions adopted are compared in Table II by means of the times for liberation from the vat ester of 20% of the sulphate originally combined. Rates of hydrolysis (in the absence of hydrogen peroxide and under nitrogen) were measured for some dyes. They are not reported but were always much lower than the rates of oxidation.

Redox Potentials

Titration curves of the form shown in Fig. 6 were obtained, obeying the equation—

$$E = E_0 + \frac{RT}{2F} \ln \frac{[\text{Ox}]}{[\text{Red}]} + \frac{RT}{F} \ln [\text{H}^+]$$

where E is the measured potential, $[\text{Ox}]$ and $[\text{Red}]$ are the concentrations of oxidised (vat dye) and reduced (leuco compound) forms respectively,

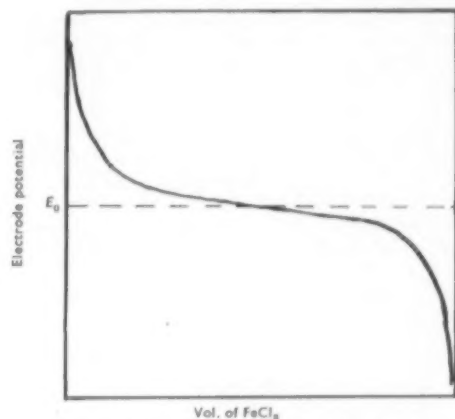


FIG. 6—General Shape of Redox Titration Curve

and $[\text{H}^+]$ is assumed to be the concentration of pyridine hydrochloride. E_0 is the standard redox potential and, at the midpoint of the titration, when $[\text{Ox}] = [\text{Red}]$ —

$$E_0 = E - \frac{RT}{F} \ln [\text{H}^+]$$

so that E_0 may be readily obtained from the midpoint potential. This was located graphically.

The standard redox potentials of the vat dyes corresponding to the vat esters listed in Table I are included in Table II, together with values for some of them obtained by Marshall and Peters².

It will be seen that some of our values differ from those of Marshall and Peters. This is particularly marked with the vat dyes derived from Soledon Orange RS (6:6'-diethoxy-2:2'-bisthionaphthenindigo) and Cibantine Brown G (bis-4:5-benzo-2:2'-bisthionaphthenindigo). Not only is there a large discrepancy between the values, but the relative orders are reversed. Since Marshall and Peters used commercial dyes and the results reported here were obtained with purified specimens, such differences are not surprising. However, when the titrations were repeated, the values for E_0 were found to be reproducible. In addition, some of the vat dyes were tested chromatographically and found to be homogeneous. It is felt, therefore, that the values reported here are more likely to be correct.

Discussion

The general shape of the rate-of-oxidation curves (Fig. 4) is similar to those obtained for the vat ester of anthraquinone-2-sulphonic acid (Part I). In accordance with the mechanism suggested in Part I, the early stage of the reaction, in which

rate increases with time, is due to the onset of direct oxidation of the ester under the catalytic influence of a slower hydrolysis. Eventually the rate of formation of catalyst (by hydrolysis) is balanced by the rate of oxidation, and subsequently the overall rate of reaction decreases as the reactants are used up. This sequence gives rise to the inflection on the rate curves. The inflection, therefore, marks the point at which the two reactions balance, so that the position of the inflection may be used as a measure of the relative contributions of the hydrolysis and oxidation reactions to the overall sequence. If the contribution of hydrolysis is the major one the inflection will occur relatively late, and *vice versa*.

It will be seen from Table II that with vat esters of Class A the inflection occurs before liberation of 10% of the sulphate originally combined, while with Class B it occurs after the liberation of 20% of the sulphate. The contribution of the oxidation reaction is therefore much more important with the Class A dyes than with the Class B. The relative importance of the hydrolysis reaction is, naturally, the reverse.

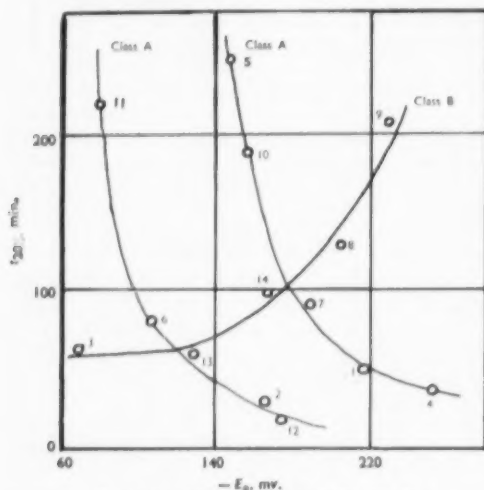


FIG. 7.—Time of Liberation of 20% of Combined Sulphate ($t_{20\%}$) plotted against Redox Potential (E_0)

This difference in the relative contributions of hydrolysis and oxidation to the total effect is further illustrated by a comparison of the overall rates of reaction with the standard redox potentials E_0 of the parent vat dyes. This comparison is made in Fig. 7, in which the times for liberation of 20% of the combined sulphate $t_{20\%}$ are plotted against the corresponding values of E_0 . Although all the dyes gave linear logarithmic plots for some part of the reaction, from which first-order rate constants k_1 could be obtained, it was felt that these constants were of doubtful significance. However, plotting k_1 against E_0 leads to the same relations as shown in Fig. 7. The Class A dyes fall into two groups, in both of which $t_{20\%}$ decreases (i.e. rate increases) as E_0 becomes more negative. There is no obvious reason for this division, but within each group the

relation between $t_{20\%}$ and E_0 is the same. With Class B dyes the inverse relation is obtained, and $t_{20\%}$ decreases as E_0 becomes more positive.

For Class A dyes, therefore, $t_{20\%}$ bears the same relation to E_0 as does σ for substituted quinones (Fig. 1), whilst for Class B the relation corresponds to that between rate constants for hydrolysis of aryl sulphates and σ (Fig. 1). Thus, both from a comparison of the position of the inflection on the rate curves (Table II) and also from the relations shown in Fig. 7 it appears that the relative importance of hydrolysis and oxidation in the development sequence varies among the vat esters.

It is not possible, because of the complexity of the dyes used in this investigation, to discuss the variation in reactivity. There is no obvious structural feature which is common to one class and not to the other, although it is perhaps noteworthy that amongst the members of Class B there is a larger proportion of dyes with substituents vicinal to the ester groups than amongst those of Class A. Steric effects may, therefore, play some part.

However, it is clear that, in order to arrive at a complete understanding of these reactions, simpler molecules, arranged in a proper series for investigation, should be used. In this connection it is significant that the simplest vat ester—that derived from *p*-benzoquinone—reacts with acidic hydrogen peroxide in two distinct stages (unpublished results of Johnson and Rahman). It appears that the ester groups are first hydrolysed by the acid, and there follows a separate oxidation of the resultant quinol. The same behaviour is shown by the corresponding monoester (*p*-hydroxyphenyl potassium sulphate). In the naphthalene series, however, the vat ester derived from 1:4-naphthaquinone and the corresponding monoester are oxidised directly with acidic hydrogen peroxide. The reactivity towards oxidation of these simple compounds is thus seen to parallel that of the corresponding quinols. The simple benzene vat ester represents the limiting case in which the contribution of hydrolysis to the development is complete. Further work in this series is contemplated.

We record our thanks to Mr. H. A. Turner, who originally suggested this work, for many helpful discussions. One of us (S.A.) is indebted to the Council and the Director of the British Rayon Research Association for a grant which enabled this work to be carried out.

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Precise Measurement of Fading on a Time-Intensity Basis

J. S. MUDD

Difficulties with *B.S. 1006* are discussed, particularly with reference to its use for testing a wider range of materials than was originally intended. It is necessary to recognise as fading changes in appearance, resulting from the action of light, other than simple destruction of colour. The precise, if arbitrary, point at which a colour may be said to have faded can be determined by means of a combined chromaticity and luminance tolerance derived from measurements in C.I.E. terms. From a consideration of their spectral energy distributions, the artificial light sources in common use for fading tests could not be expected to give results comparable with those obtained with natural daylight, but the light from a xenon arc is shown to have a similar effect to daylight. A simple type of fading lamp is described using a 150-watt xenon lamp. Since the light falling on a pattern can be maintained at constant intensity for a considerable time and the intensity can be accurately measured, the fastness to light of such a pattern can be precisely expressed in terms of the amount of light (in kilo-ft.-candle-hr.) required to fade the pattern to a predetermined colour tolerance.

Referring to the problem of the assessment of fading, the following passage appears in a paper presented to the International Conference of Leather Chemists Societies at Barcelona in 1953¹: "It is probable that until more precise methods of determining fading are perfected, based on colorimetric determinations, the 'blue wool' scale, devised by the Society of Dyers and Colourists, will be generally used." In a paper by Vickerstaff and Tough² in 1949 the statement was made that: "It may be possible in the near future to define light fastness in some such manner as the following—The light fastness of a dyeing is to be measured in terms of the exposure in lux-hours which is required to produce a change of ten just perceptible steps in the colour of the pattern." The possibility of linking fading with a system of agreed colour tolerances was also suggested by Mudd and Downs³, but there were two problems which at that time seemed very difficult to solve. One was the need for a much clearer definition of colour tolerance based on practical working conditions. The other, and more difficult, problem was to find an artificial light source which would cause fading in a similar manner to daylight, but free from the continual variation in intensity and energy distribution associated with the natural illuminant, so that determinations could be made on a time-intensity basis. Personal experience as well as many reported cases⁴ indicated the difficulty of correlating the results of fading in natural daylight with fading caused by artificial light sources such as mercury-vapour and carbon arc lamps. J. McC. C. Boswell, in a private communication, has recently reported that "as far as a series of commercial leathers is concerned there is no useful relationship between fading to daylight and ultra-violet light." There is also a warning about the use of artificial light sources for fading tests in *B.S. 1006*⁵, differences in results being attributed to differences in the emission spectra of the fading lamps. Differences can occur also with daylight fading tests under varying lighting conditions at different times of the year⁶.

VISUAL METHODS OF ESTIMATING FADING

Consider first the system of visual assessment of fading in general use at present, which is incorporated in *B.S. 1006*⁵, the blue wool scale devised by the Society of Dyers and Colourists. It is officially described as a method for the determination of fastness to daylight of coloured textiles,

and possibly if its use is restricted to this somewhat limited field it is entirely satisfactory. Perhaps not entirely satisfactory, for there are differences of opinion between individual operators in the assessment of fading and there is the rather ambiguous use of alternative fading figures. The use of this method has been extended to the determination of the light fastness of many materials other than textiles, and its exponents claim that it is equally useful in these fields, although it is asking rather a lot of the human eye, when making an assessment, to eliminate all considerations of chromaticity, luminance, texture, and gloss and to concentrate solely on an estimation of contrast ratio. One can understand its ready appeal to the textile industry, but it is also easy to appreciate the apprehensions of a paint chemist who is asked to compare the fading of a red enamel with that of a blue wool pattern, even if, as we are told, these apprehensions are unfounded. In spite of these difficulties, whether imaginary or real, *B.S. 1006* undeniably gives a more useful assessment of the fastness of the red enamel than could possibly be estimated from the obsolete madder scale. This is a scale which has only one reference point, and that ill defined, namely madder lake in a linseed-oil paint film, which is assumed to have a light fastness of 10, with other pigments arranged in descending order, a system obviously liable to subjective errors of considerable magnitude. The fact that materials varying considerably in light fastness can result from the same pigment in different media or mixed with white pigments has destroyed once and for all the idea that to any particular pigment can be assigned a definite fading figure. It is now generally accepted that such a fading figure applies only to the particular pigmentation or dyeing for which it was determined. A further development is the recognition as fading of colour changes other than simple destruction of colour, such as those occurring when yellow patterns become orange on exposure, or red patterns become bluer. Of particular note in the case of pigmented coatings are the lead chrome pigments, which exhibit very little change in chromaticity, but darken rapidly. These were described under the madder rating as having a light fastness of 10, i.e. as equal to madder lake, but with the qualification "darkens on exposure", with no indication whatever of the exposure required to produce a drastic alteration in appearance. It seems reasonable that, for the purpose of assessing

the fastness to light of coloured materials, fading might be defined simply and unequivocally as an alteration in appearance consequent on exposure to light.

There are other difficulties, however, in using *B.S. 1006*, and one of these is concerned with the precise stage at which a material may be said to have faded. In practice there might be very considerable fading before the effect was noticed. For example, with a curtain material it might depend on the keenness of a house-proud woman to detect a change in colour, or the keenness of a visitor to make remarks that are often better left unsaid. But in fact the stage can only be that at which the part which has been exposed to light shows a visual difference from the original. The necessity of fading to a common point, even if it is not the right point, has been recognised by the inclusion in *B.S. 1006* of the grey scale, which is, in effect, only a method of determining changes in luminance. This is obviously desirable if differences in assessments between various operators are to be avoided. There are many cases, however, in which luminance changes are only of secondary importance compared with changes in chromaticity. Such changes are difficult to interpret in terms of *B.S. 1006*. In the case of lead-chrome pigmentations the change is almost entirely one of luminance, but in the reverse direction to normal fading. A slight darkening of chrome yellow is often confused with greenness, quite naturally, since mixtures of yellow and black pigments produce a range of pseudo greens.

USE OF COLOUR MEASUREMENT

Visual assessment of small colour differences is admittedly difficult, and differences of opinion frequently occur even amongst so called "trained colourists", but such colour differences can be determined colorimetrically and recorded in C.I.E. terms. It is even more difficult to assess visually the precise point at which a pattern may be said to have changed colour, but this point can be found easily by extrapolation from colour-measurement data.

If it is accepted that small colour differences may properly be determined by colour measurement, then it follows that one may use a colour tolerance, which combines the effects of changes in chromaticity and luminance, representing a just noticeable difference under normal operating conditions, as the point of change in determinations of fading. A method of arriving at a colour tolerance or *T* figure, based on liminal differences under normal lighting and viewing conditions, as distinct from the refined conditions encountered in making photometric observations for purposes of fundamental research, has been evolved by a committee of the Society of Leather Trades Chemists⁷, and it is suggested that this might form the basis of a precise method of determining fading, the answer, in fact, to the first of the two problems mentioned at the commencement of this paper.

DETERMINATION OF COLOUR TOLERANCES

In the S.L.T.C. official method of determining colour tolerances the standard chromaticity

figures *P* of the test pattern are plotted in the appropriate position on a chromaticity chart, and the graphical tolerance in the form of an ellipse is drawn about this point (Fig. 1). Major and minor axes and the slope of the major axis of the ellipse are found from a table. On the same chromaticity chart is plotted the point *S* representing the pattern that is to be compared with the test pattern, or equally representing a portion of the test pattern itself that has been exposed to light.

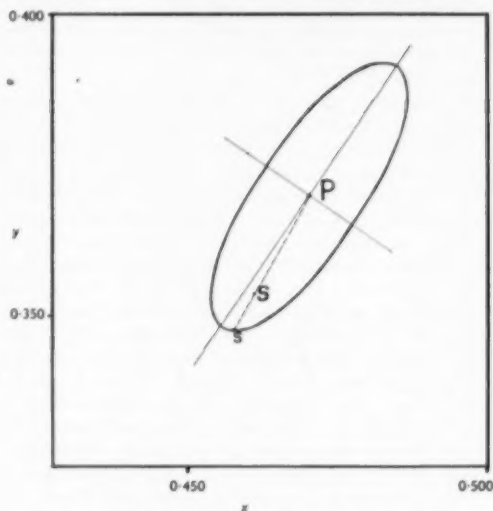


FIG. 1—Determination of Chromaticity Tolerance

The deviation of the second chromaticity point from the standard is expressed as a percentage of the permitted tolerance and recorded as *C_t*. In Fig. 1—

$$C_t = \frac{PS \times 100}{P_s}$$

A metrical tolerance for the luminance factor β is also computed for the test pattern. This tolerance varies from 0.2 at $\beta = 0$ to 1.0 at $\beta = 100$ (Fig. 2).

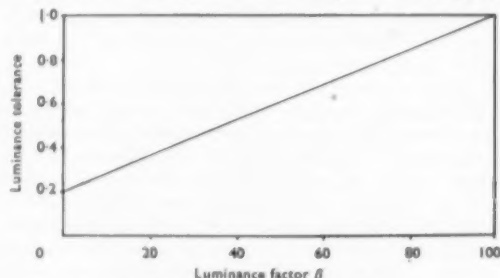


FIG. 2—Determination of Luminance Tolerance

The variation in luminance is expressed as a percentage of the permitted luminance tolerance and recorded as β_t . The combined tolerance is then given by—

$$T = \frac{\sqrt{C_t^2 + \beta_t^2}}{100}$$

It must be clear that, whereas by visual examination it is virtually impossible to stop the fading at

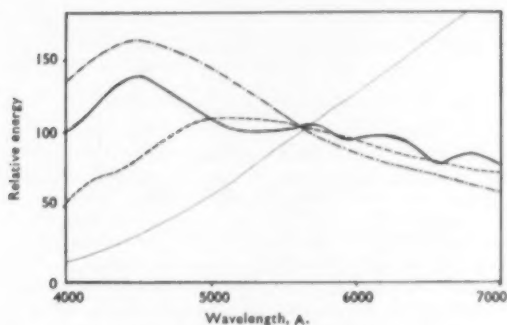
a predetermined point, it is quite easy by colour measurement to determine the exact point, in terms of time of exposure, at which $1.0 T$, $2.0 T$, or xT is reached.

CAUSES OF ANOMALOUS FADING

In spite of McLaren's contention⁸ that differences in relative humidity are the principal causes of anomalous fading with different light sources, it is unlikely that wide differences in energy distribution can be disregarded. In a later paper⁹, however, he says, with reference to the Standard Fading

Hour concept, relating arc-lamp to daylight exposure: "Now that the importance of the spectral composition of the light is known, it would appear that this system contains inherent defects."

It has already been mentioned that daylight varies both in intensity and in spectral energy distribution according to latitude and prevailing weather conditions. Indeed, it is difficult to define "average" daylight, but Fig. 3 shows the relative energy distribution of (i) Mid-June sunshine at Washington¹⁰ and (ii) North skylight¹¹. Fig. 3 shows also (iii) the distribution for tungsten lighting (standard illuminant A), and Fig. 4 those for a typical carbon arc¹² and for a low-pressure mercury-vapour lamp¹³. For comparative purposes the relative energy in each case has been adjusted to the same value at 5600 Å. None of these artificial sources is in any way comparable with daylight, and one would expect considerable differences in fading if patterns were to be exposed to similar quantities of each of these types of light. Fig. 5 shows the relative energy distribution of the standard international illuminants B and C, but, as these are produced by heavy filtering from tungsten sources, it is impracticable to produce them in the intensities required for fading tests.



- (i) --- Mid-June sunlight at Washington, D.C.
- (ii) North skylight
- (iii) - · - · Standard illuminant A (tungsten-filament)
- (iv) — High-pressure xenon discharge lamp (type FA 5)

FIG. 3—Relative Energy Distributions of Xenon Arc Light, Daylight, and Tungsten-filament Light

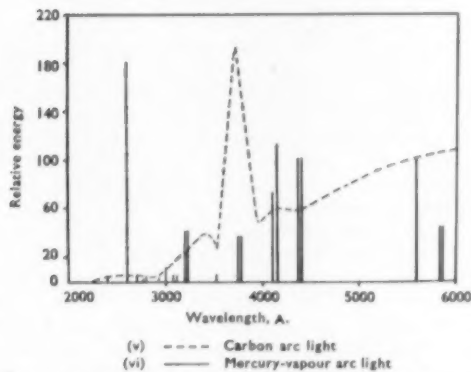


FIG. 4—Relative Energy Distributions of Carbon and Mercury Arc Lights

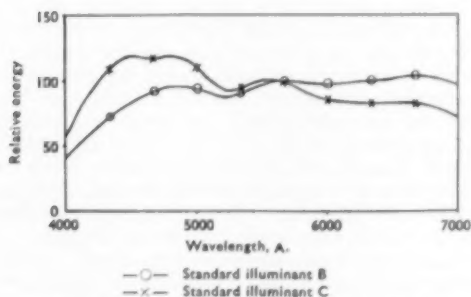


FIG. 5—Relative Energy Distributions of Standard Illuminants B and C

HIGH-PRESSURE XENON DISCHARGE LAMP

Fig. 3 shows the relative energy distribution of (iv) a high-pressure xenon discharge lamp (Type FA 5 manufactured by the British Thomson-Houston Co. Ltd.)¹⁴ superimposed on the daylight curves. From a consideration of this curve it is reasonable to assume that such a light source will have a fading action similar to, if not identical with, that of natural daylight. If a coloured pattern is progressively faded and the results are plotted in C.I.E. terms on a chromaticity chart, they rarely follow a straight line joining the point representing the original colour and the white point, but tend to take a devious route. Patterns of red, green, and blue surface-coating colours were progressively faded both by natural daylight and by artificial (xenon) light. The chromaticity changes are shown in Fig. 6-8. They indicate that, for all practical purposes for fading tests, natural daylight and artificial (xenon) light have the same effect, with the important difference that, whereas daylight is inconstant and difficult to assess quantitatively, the artificial source is constant and available over long continuous periods, and its intensity can be accurately determined. If, therefore, the use of a xenon lamp in a suitable instrument is the answer to the second part of the problem, it becomes possible to fade a pattern to a tolerance limit which represents a definite colour change, and to determine, in recognised units, the quantity of light required to cause that change. The unit in this case is the foot-candle-hour, but in view of the quantities of light required for normal fading tests the kilo-foot-candle-hour (k.f.c.h.) is more generally useful.

It is suggested that the most informative and generally useful method of describing the fastness to light of a coloured material is in terms of the number of k.f.c.h. of a standard light source, sufficiently close in its behaviour to be used in

place of natural daylight, required to produce a change of 1.0 T on the S.L.T.C. scale, that is a just noticeable change in appearance. Such a method has certain obvious advantages, but perhaps the greatest of all is the use of a continuous scale. A scale of eight fixed points may be adequate to describe the fastness of textiles but not the diversity of coloured materials about which information is required. For example, most commercial finished leathers lie between 4 and 5 on the *B.S. 1006* scale. The individual differences between these leathers are significant and could and should be much more clearly defined. A number of materials, including spirit lacquers and some of the more fugitive dyeings on paper and leather, are

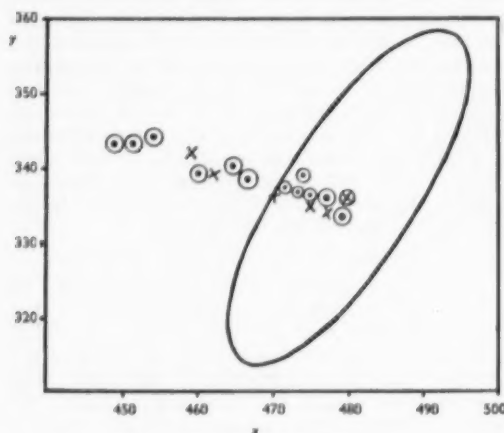


FIG. 6—Red Pattern

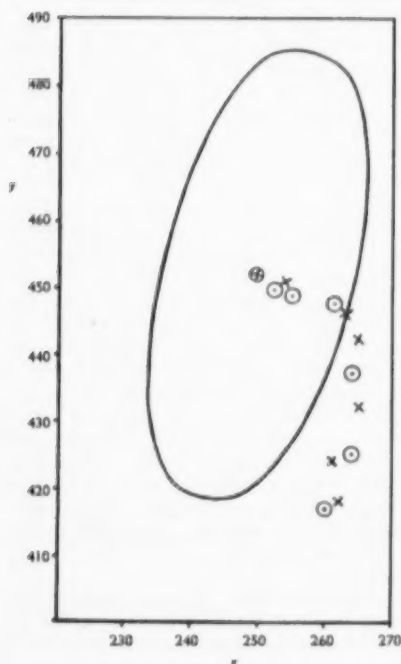


FIG. 7—Green Pattern

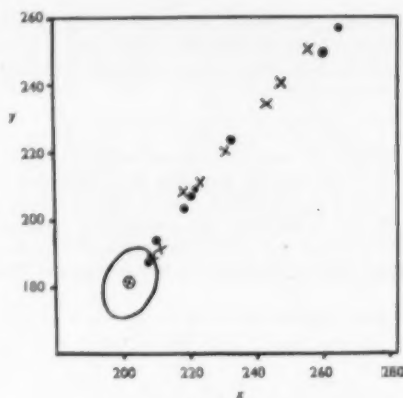


FIG. 8—Blue Pattern

x Daylight
o Xenon light

FIG. 6-8—Chromaticity Changes of Red, Green, and Blue Patterns in Daylight and in Xenon Light

grouped together in the category "less than one", yet individually one may be several times as fast as another. Small differences in light fastness due to changes in technique may be significant, even if they are not sufficient to lift a material from one *B.S. 1006* grade into another.

PRACTICAL ILLUMINATION INTENSITIES

Some tests carried out in a room in a country house showed great variation in the intensity of the illumination. The maximum in a window facing South and falling on the curtains at 2 p.m. on a bright day was 7,200 ft.-candles. Although the sky continued cloudless, this fell to 2,600 ft.-candles by 5 p.m. On a dull day the maximum was only 610 ft.-candles. The corresponding intensity which would fall on a book or a paper measured 200, 100, and 20 ft.-candles with the observer seated. Under the same conditions the light falling on a number of pictures varied from 65 down to 1 ft.-candle, the average illumination probably being not more than 10 ft.-candles. In artificial light, although the room is regarded as being adequately lit, the intensity on seven pictures varied from 0.4 to 2.5 ft.-candles, and the maximum intensity for reading was 9 ft.-candles. Such experiments at least give some guide to the translation of a precise fastness figure into practical terms.

A fading figure for a curtain material of 4,000 k.f.c.h. means that no significant change will occur in something like 800 hr. of direct sunshine, which is more than most windows receive in England in a year. On the other hand, the dyes used in integral tripack processes for colour photographs on paper are only of the order of 30 k.f.c.h. fastness, but these prints can be hung for a long time in a room where the average illumination on the pictures is less than 10 ft.-candles. Another advantage is the avoidance of errors due to variations in assessment by individuals, against which must be set the possibility of instrument variations. It must be remembered that the position today with regard to instruments for colour

measurement is very much better than it was when the work on fastness to light was initiated by the S.D.C. Modern photoelectric colorimeters not only are sufficiently accurate for colour measurement for fading tests but are sufficiently rapid in operation to be used for routine control. With the figures for the reference standard already known, the time required to measure a second pattern and arrive at the tolerance figure has been shown in the author's laboratory to be just under 4 min. The only exposure necessary is that of the material to be tested, and results can be directly compared with others made independently or at different times. The advantage of having a light source of considerable intensity available for long continuous periods has already been indicated.

EXPERIMENTAL FADING LAMP

For the purpose of the experimental work described in this paper an F.A. 5 xenon lamp was fitted in the instrument shown in Fig. 9. A condenser lens system collects light from the lamp over a solid angle of approx. 90°. This light is passed through a collimating lens to form a uniform patch of light 1 in. in diameter on a pattern placed about $\frac{1}{2}$ in. in front of the lens components. Four of these systems are operated from one lamp, so that four patterns can be exposed at any one time.



FIG. 9—Experimental Fading Lamp

The lamp envelope and the glass lens components remove any radiation below about 3200 Å. Under actual operating conditions not only is the radiation similar to daylight in the visible region of the

spectrum, but the energy distribution in the ultraviolet and infrared regions is of a similar order. Although the intensity of the light falling on the test patterns is of the order of 4,000–5,000 ft.-candles, it has not been found necessary to use a heat filter. Usually when the lamp power is greater than 150 watts a heat filter is necessary to avoid high ambient temperatures. The F.A. 5 lamp is essentially a heavy-current low-arc-voltage device consuming about 10–12 amp. with an arc voltage of 15 v. As the pressure of the xenon gas in the lamp is above atmospheric, the breakdown voltage across the arc gap is high, approaching 3,000–4,000 v. It is therefore necessary to use a pulse voltage to start the lamp on a supply voltage of 250 v. A.C. For laboratory work it is convenient to use two M.R.G. 101 (B.T.H.) chokes in parallel and in series with the lamp, using a Tesla spark coil as a simple way to start the lamp. With a choke ballast circuit the light output changes with variations in the supply voltage. With a 5% change in mains voltage the light intensity changes by approx. 12–15%, so that where the supply voltage is unstable it is desirable to have some form of automatic voltage control. The normal lamp life of the F.A. 5 is quoted by the makers as 500 hr., but the author has run one for 2,000 hr. with only a 20% drop in light intensity. The light intensity at the surface of the test patterns can be checked by a normal type of photometer using a selenium cell in conjunction with a neutral density filter, but the instrument requires to be first calibrated against a xenon standard.

The figures given in Table I show the progress of the fading of a green surface-coating colour, actually a Brilliant Green—polymolybdotungstic acid lake reduced with Titanium White in a casein medium, measured according to the S.L.T.C. tolerance system. The corresponding progress of fading in natural daylight is interesting, but of course the amount of light involved in one hour of daylight fading varies considerably, and the results are not quantitative.

Fig. 10 shows the method of plotting so that the precise point at which a colour difference of T is reached can be determined. It is clear that in this case T is reached in 8 k.f.c.h.

Many other patterns, including a number of commercial leathers, have been examined in this

TABLE I
Fading of a Green Pattern in Xenon Light and in Daylight

Xenon (k.f.c.h.)	Daylight (hr.)	β	x	y	C_t	β_t	T
0	—	16.8	0.250	0.452	—	—	—
2.5	—	16.7	0.253	0.450	28	33	0.43
6.1	—	17.0	0.255	0.449	49	67	0.83
12.3	—	17.0	0.262	0.447	114	67	1.32
31.9	—	17.1	0.264	0.437	153	100	1.83
80.9	—	17.3	0.264	0.425	183	167	2.48
—	0	17.0	0.250	0.452	—	—	—
—	0.5	17.0	0.254	0.451	35	0	0.35
—	6.5	17.3	0.263	0.446	124	100	1.59
—	66	17.3	0.265	0.432	171	100	1.98
—	210	17.6	0.261	0.424	189	200	2.56

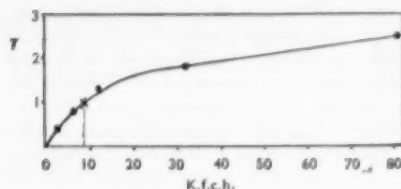


FIG. 10—Determination of Fading Figure

way, and the results have shown general agreement with the known reaction of similar materials to daylight. In one case a pink leather, condemned as fugitive on a carbon-arc exposure, was found to be reasonably fast to the xenon light, and this was confirmed by subsequent daylight exposure.

Fading tests were also made, according to the method described above, on some of the blue wool patterns of B.S. 1006, with the results shown in Table II.

TABLE II
Fading in Xenon Light of Standard Blue Wool Patterns (B.S. 1006)

Standard No.	K.f.c.h. required to fade to <i>T</i>
1	4.6
2	8.2
3	18.0
4	156
5	600
6	1900
7	2500

It is obvious that these results do not agree with the spacing of the blue wool patterns found by Ricketts¹⁵, and there are two main reasons for this, apart from the subjective errors possible with visual assessment. Firstly the point at which fading is examined is important, because rarely, if ever, does fading proceed at a constant rate, and if the patterns in Table II had been faded to a much greater extent than is represented by *T*, the ratio between them would have been different. The author contends that, as *T* represents a noticeable difference in appearance between patterns under normal viewing conditions, when the material reaches this point it has definitely faded. If, however, it is really necessary to define a point representing "very bad fading", this can also be done in precise terms. For example, B.S. 1006 refers to fading to a contrast of grade 3 on the Geometric Grey Scale, which would be equivalent to 7.5 *T*. The fading point has to be an arbitrary one, but however this is selected, it should at least

be an exact point defined by colour measurement and not by visual estimation. Secondly, the results given by Ricketts were derived from exposures to carbon arc light, which is very rich in ultraviolet radiation compared with natural daylight, and would not be expected to correspond with exposure to daylight or a xenon arc. McLaren⁹ has shown that the faster blue wool standards are proportionally more affected by such light than the relatively fugitive ones. Fading to light from a carbon arc would therefore be expected to result in a compression of the scale of ratios, which is evident from an examination of Ricketts' results.

DEVELOPMENT WORK

It is to be hoped that this xenon lamp will be developed further, with improved methods of control and starting. Higher intensities could be employed in larger fading lamps than the one described. Many more fading tests have to be made and the work is continuing, but it is hoped that the foregoing will show the possibility of a system of precise measurement of fading on a time-intensity basis.

* * *

In conclusion the author wishes to express his appreciation of the help given by the British Thomson-Houston Co. Ltd., and particularly by Mr. E. Beeson of their Lamp Research Department in the construction of the prototype fading lamps.

THE LABORATORY

NENE FINISHES (NORTHAMPTON) LTD.
NUNN MILLS ROAD
NORTHAMPTON

(Received 20th August 1956)

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EXPLANATORY PAPER ON MODERN THEORY

Colour Perception

H. W. ELLIS

The sensation of colour is of fundamental importance to everyone who reads this *Journal*, and is of fundamental interest to nearly every human being. Like many other vitally important things, however, it tends to be taken for granted by the majority of people without in any way being understood. Naturally, those connected with the colour-using industries have at least some knowledge of the principles governing colour, but these principles belong to the realm of physics, whereas most dye-users have had a chemical training. For some reason, it seems that, for many people, there is a great gulf fixed between these two branches of science; hence they may spend their lives dealing with colour in one way or another without ever having any basic knowledge of the sensation which they give so much time and employ so much skill to produce. Of course, it must at once be admitted that such basic knowledge is not essential to the proper carrying out of their jobs, but it is upon the assumption that a simple exposition of the physics of colour should be of interest and value to all colour-users that this paper (the author of which is a chemist) is written.

Colour is a sensation, not an objective reality. The use of the word "colour" as a substantive is so universal, however, that this fact tends to be overlooked. No object possesses colour in its own right: no colour exists in a completely dark room. The reality is that colour is a psychophysical sensation, depending upon both a physical stimulus in the eye and a psychical response in the brain: if either of these factors is absent, the observer is unaware of colour, and in the absence of such awareness there can be no evidence of the existence of colour. This statement may seem obvious when read, but it states a fact which is very often overlooked.

The objective study of colour is, therefore, concerned with two factors—the nature of the stimulus causing the sensation of colour, and the means by which this stimulus produces the sensation. To begin with, we all know that the stimulus is "light". Light consists of electromagnetic radiations of the same nature as radio waves but of considerably shorter wavelength. Radiation of wavelength between approx. 0.4μ . and 0.75μ . ($1 \mu = 10^{-3}$ mm.) will stimulate the eye and cause the sensations of light and colour. Shorter or longer waves will not stimulate the eye and so cannot be described as light, although in common usage radiations from about 0.2μ . to the threshold of visibility at 0.4μ . are often referred to as "ultraviolet light"; the term is, however, a misnomer.

Radiations consisting only of narrow bands of wavelengths also give rise to the sensation of colour, starting with violet at the shortest visible wavelength and proceeding through the well known

order—violet, blue, green, yellow, and orange—to red at the longest visible wavelength. This series of colours is, of course, known as the *visible spectrum*, and the distribution of colours in it is shown in Fig. 1. The different colours merge imperceptibly into each other, but for the majority of observers the colour zones in Fig. 1 are quite well defined.

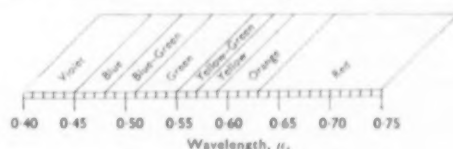


FIG. 1—The Colours of the Spectrum

Ordinary white light, such as sunlight or light from an incandescent-filament lamp, consists of a mixture of all the wavelengths in the visible spectrum (as well as both shorter and longer wavelengths). When a beam of such light passes through a glass prism, it is refracted at an angle depending upon the wavelength, and the constituent radiations in the beam are therefore spread out. If allowed to fall upon a white surface, they can be seen separately in the spectrum order, the red waves being deflected least and the violet waves most.

The sensation of colour arising from a narrow band of wavelengths is the brightest aspect of that colour which can be perceived, and is known as a *fully saturated colour*. If light of the remaining spectral wavelengths is added to the beam in small amounts, the resulting sensation of whiteness dilutes the colour, or *desaturates* it, and in common parlance the colour becomes paler (e.g. bright red to pink). If, on the other hand, two narrow bands of wavelengths are mixed, an intermediate hue is seen, which can sometimes be almost as fully saturated as a single spectral colour. For example, admixture of a spectral red with a spectral green will produce the sensation of a yellow only a little less saturated than a spectral yellow. The addition of a third wavelength to the mixture will, however, usually tend to desaturate it and so reduce the colourfulness of the resulting sensation.

So far, we have dealt solely with coloured light, which is the term loosely used for visible radiations which are either monochromatic or consist largely of one or two fairly narrow bands of wavelengths. Although it should again be mentioned that the sensation of colour can be aroused only by a stimulus of this nature, to the vast majority of colour-users the source of colour sensation is not a spectrum or a self-luminous body, but a coloured surface. Naturally, such a surface can be seen only by means of the light which it reflects, and, as is well known, a surface appears coloured, even though illuminated by white light, because it has

the property of absorbing some of the wavelengths which fall upon it and reflecting the remainder to a greater or lesser extent. Whereas the colour sensation produced by a painted surface or a dyed textile fabric is still the result of a mixture of radiations entering the eye, the factors governing the resulting hue are quite different from those so far mentioned.

In dealing with the results obtained by mixing dyes or pigments, it must always be remembered that their colour is produced by *subtracting* some of the incident light and reflecting the remainder. Thus, the effect upon colour of a given dye depends upon the wavelengths which it absorbs rather than those which it reflects. From this it follows that, whereas the admixture of three (or more) beams of light of different wavelengths tends to produce a predominantly white sensation, the admixture of a corresponding number of dyes absorbs more and more of the incident light, the amount reflected becoming progressively less; the sensation produced thus tends towards black.

PRIMARIES

All colour-users are familiar with the term *primary colours*, by which is normally meant those colours which cannot be produced by the admixture of two or more different colours. The true primaries are those which are concerned in the mixing of "coloured light" or, properly speaking, in additive colour mixing. They are red, green, and blue, and although the actual wavelengths corresponding to these colours are not highly critical, it has been found by experiment that a red of wavelength 0.65μ , a green of 0.53μ , and a blue of 0.46μ are typical values. The primary colours normally used by dyers, on the other hand, are generally described as red, yellow, and blue. To understand this discrepancy it must be remembered that the mixing of dyes controls colour by a subtraction process, and that, as already stated, a dye controls the final hue by virtue of the wavelengths which it absorbs. The true *subtractive primaries* are, therefore, those colours which are produced by subtracting each of the additive primaries—red, green, and blue—from the complete spectrum of white light. Accordingly, "minus red" is the subtractive primary produced by absorbing all red wavelengths and reflecting the remainder, and is blue-green (cyan) in colour; minus green is magenta; minus blue is yellow. Cyan, magenta, and yellow are therefore the true primaries used in dyeing. It is possible, and in fact is common practice, to use a "neutral" blue and red in place of cyan and magenta, and the reason for this is that, generally speaking, it is not possible to obtain a dye which will totally absorb radiations of a certain group of wavelengths and reflect all others completely. Thus, each dye in a three-colour mixture will control not only its own principal absorption band but also, to a lesser extent, the absorption bands of the other components. Hence the final colour will inevitably be somewhat darker than would have been the case were dyes with ideal absorption curves available, and the diminution in efficiency caused by using blue instead of cyan, and red

instead of magenta, is therefore unnoticed. In addition, many modern dyes are comparatively dull in colour, i.e. they absorb fairly considerably in other wavelengths than that of their minus colour, and hence their use as "primaries" is not very efficient in that bright mixture dyeings cannot be produced owing to this overall absorption.

COLOUR VISION

Of equal importance in the study of colour to a knowledge of the stimulus which gives rise to a colour sensation is an understanding of the manner in which this causation operates, or in other words how, through the medium of the eye, a colour sensation is made to occur in the brain. A very great deal of painstaking and delicate research has been undertaken on this subject, mainly during the past hundred years, and although much remains to be discovered, many aspects have been made clear.

It is well known that, when light falls upon the retina, which is the light-sensitive surface at the back of the eye, impulses are transmitted through the numerous fibres forming the optic nerve to the brain. The actual light-sensitive elements in the retina are known as the *rod* and *cone* receptors. The distribution of these varies across the retina: the central area, known as the *fovea*, which subtends an angle of about 2° of the visual field, and which is the area of maximum resolution, is populated entirely by cones—some 100,000 of them—but with increasing distance from the fovea the proportion of rods increases steadily, until at the peripheral zone the retina is populated entirely by rods.

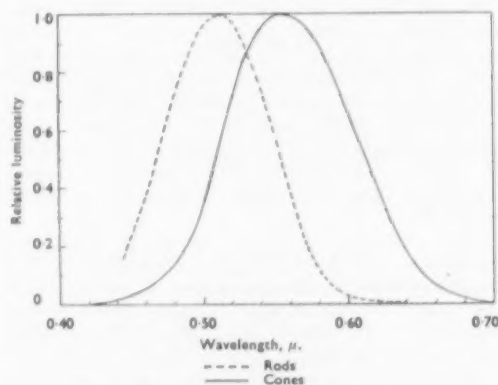


FIG. 2—Spectral Sensitivity Curves of the Eye

The cones are the receptors which operate at high levels of light intensity, while the rods operate at low intensities. Only the cones can cause colour sensation; the rods are completely "colour-blind", and can give rise only to monochromatic vision. This explains why, in a nearly dark room, it is impossible to distinguish colour; we are accustomed to think that "it is too dark to make out the colours", whereas the fact is that, at levels of illumination too low to stimulate the cones, vision is mediated solely through the rods, which, although very sensitive to light, are not

sensitive to that quality of light which we call colour.

The sensitivity of both rods and cones to light varies with wavelength, and this is illustrated in Fig. 2. As might be expected, the sensitivity is lowest at either end of the spectrum and rises to a maximum between. The cones are most sensitive to light of 0.555μ , which is greenish yellow in hue, while the maximum sensitivity of the rods is to light of 0.510μ , which is green. This explains why green light is used when developing highly sensitive panchromatic photographic materials: these are sensitive to all wavelengths, and only the minimum intensity of light can be allowed. Since at very low intensities vision is mediated almost entirely through the rods, visual sensation can be obtained with the lowest possible amount of light if this is of the wavelength to which the rods are most sensitive.

It must be understood that the foregoing remarks refer solely to the *sensitivity* of the eye to varying wavelengths of light and do not refer to colour sensation. We are all aware of this varying sensitivity, although we do not usually think of it as such. Instead, we say that yellows and greens are very "light" colours, and that reds and violets are "dark".

An interesting phenomenon occurs when the intensity of illumination is fairly low, but high enough partially to stimulate the cones as well as the rods. In such a case, the difference in spectral sensitivity of the two types of receptors becomes very noticeable and causes what is known as the *Purkinje effect*. From Fig. 2 it will be seen that the rods are far less sensitive than the cones to reds and oranges, and therefore when a considerable proportion of the total visual sensation is being mediated through the rods, red or orange surfaces appear much darker in proportion to other colours than they do in strong light. This effect can easily be seen in a garden (in summer) at "lighting-up time" or thereabouts. Red roses or orange marigolds appear very dark, although there is still enough response from the cones (which, it will be remembered, are the only source of colour sensation) to enable the colour of the flowers to be distinguished. By contrast, the green grass appears lighter, owing to the extra sensitivity of the cones to green light.

TRICHROMATIC VISION

The actual mechanism by which the eye transmits nerve impulses which differ by that quality which we describe as colour has never been fully elucidated. The generally accepted theory is that there are three types of response from the cone receptors, and that the whole range of colour sensations is produced by variations in the relative magnitude of these responses. The principal arguments in support of this theory are the facts that almost any colour can be produced by suitable admixture of the three primaries red, green, and blue, and that the sensation of the primaries themselves cannot be produced by any admixture of other colours, whether primaries or not. It could,

therefore, reasonably be postulated that there are three types of cone receptors (although no apparent differences have yet been established) each of which, though sensitive to a wide range of the spectrum, possesses maximum sensitivity to a wavelength corresponding to one of the primaries. A given radiation will accordingly promote responses from each type of receptor, of varying magnitude according to the sensitivity of the receptors to the colour, and the resulting sensation will be produced by the integration in the brain of these different responses.

COLOUR PERCEPTION

The foregoing remarks on colour vision are objective and mainly factual. The sensation of colour is, however, psychological as well as physical in nature: impulses from the eye, transmitted through the optic nerve, are perceived or interpreted by the brain. This interpretation seems not always to be logical, or to fit in with known facts about the stimulus which is being construed. Thus, a given stimulus in terms of intensity and wavelength should always produce the same sensation, but actually this is not so. It would appear that this is due partly to a "pre-knowledge" of what the sensation should be, and partly to the effect which contrasting surroundings have upon the appearance of an object. For example, suppose that a white-painted object is observed in a room illuminated by the light of ten lamps. If five lamps are then extinguished, the object still appears to be white and not grey, even though it now reflects, and the eye receives, only half the intensity of light that it did formerly. This is because we are conscious of a lower level of illumination of the whole room, and not solely of the object. For the same reason, a coloured surface does not appear to change colour under reduced illumination, so long as this is not so low as to allow the Purkinje effect to occur. On the other hand, if the amount of light reflected from the object only is reduced, the general illumination level remaining constant, the visual sensation undergoes a considerable change. This is a most interesting comparison. It will readily be appreciated by any colour-user that if the white-painted object—which remained white in appearance when the amount of light reflected from it was halved by reducing the general illumination level—is repainted with a mixture of equal parts of white and of a perfectly neutral black, it will then appear grey. When illuminated by the original ten lamps, the light reflected from it into the eye is identical in amount and quality with that reflected from the white object under five lamps, but the appearance is strikingly different. This phenomenon illustrates the effect which contrast (and particularly a variation of contrast) has upon colour perception.

These illustrations serve to emphasise that colour perception, and indeed all visual sensation, is a psychophysical process, and while the physical aspect can be dealt with objectively, and, to a large extent, can be controlled quantitatively, the psychological sensation can play strange tricks—as every colour-matcher knows.

DEFECTIVE COLOUR VISION

So-called "colour-blindness" is a defect which normally should not affect readers of this *Journal*. A few facts about it should, however, be of interest. In almost all cases, colour-blindness is congenital and is transmitted in such a way that it occurs far more frequently in men than in women, although the female child of a colour-blind parent can be a carrier of the defect and pass it on to her son, though possessing normal colour vision herself. The incidence of one or other form of defective colour vision among males is about 8%, while the figure for females is probably less than 0.5%.

There are three main types of colour-blindness, known as anomalous trichromatism, dichromatism, and monochromatism. As may be inferred from these terms, they refer to the number of primary stimuli to which the individual is sensitive, and while the facts concerning them would appear to confirm the existing theory of trichromatic vision, it must be remembered that it has so far not been possible to confirm this by actual experiment.

Anomalous Trichromatism

In this form of defect, the observer, though sensitive to all three primary stimuli (red, green, and blue), has reduced discrimination in one of them, and hence will make matches which will differ from those made by normal observers, particularly if the colour to be matched contains a large proportion of the primary to which his sensitivity is reduced. His recognition of colours will not, however, differ significantly from normal, and in the majority of cases his defect will not be particularly noticeable, unless his activities should include the exact matching of colours. Anomalous trichromats are the most numerous among colour defectives, and account for about 5.5% out of the total of 8% mentioned above.

Dichromatism

This is a more serious form of defective colour vision, and is characterised by the fact that the observer can match any colour—satisfactorily to himself—with only two primary stimuli instead of three. In all except a tiny proportion of dichromats there appears to be no sensation of colour-change in the spectrum from red through orange to yellow and green. Hence the observer can make a match which satisfies him by using blue and any one other radiation of wavelength longer than green. Thus, the sensations he experiences by mixing blue with yellow and blue with orange will be similar in hue, though perhaps different in luminosity. Hence he will confuse greens with browns and fawns. He will also, of course, confuse all bright spectral colours in the yellow-orange-red region.

In addition to this lack of discrimination, about half of all dichromats possess abnormally poor sensitivity to red light, viz. to radiations of 0.63μ , and longer. Reference to Fig. 2 will show that, for the cone receptors, the relative luminosity of radiation of this wavelength is about 0.3, but for the defective observer the figure will be of the order of 0.05, or only about one-sixth. Since, therefore,

he has no sensation of hue, such an observer can easily confuse a full red with black, owing to his low sensitivity to red radiations, and the possibility of his attending a funeral in a red tie (if he be single) is therefore a real one.

The very small proportion of dichromats who do not suffer from the "red-green blindness" which has just been described are those whose discrimination is normal in the red-green part of the spectrum, but is absent in the violet, blue, and blue-green region. This form of defect is so rare, however, that detailed information about it is lacking.

From the fact that a dichromat can match any colour with only two primary stimuli, it can be deduced that his defective colour vision is due to the fact that only two types of cone receptors exist in his retina instead of the normal three. This is, at present, only surmise, but it does fit the known facts, and is certainly reasonable.

Monochromatism

This defect is, fortunately, very rare. As its name implies, the observer has no hue discrimination whatever: the spectrum is a monochrome, and the only difference he can see between colours is one of lightness. It would appear that this defect is very probably due to a complete absence of activity of the cones, and that vision is mediated solely through the rods, which are insensitive to colour differences. This theory is supported by the fact that nearly all of the few monochromats who have been examined are hypersensitive to light, which is to be expected when it is remembered that the rod receptors are highly sensitive to low levels of illumination. In addition, it will be remembered that the retinal area known as the fovea contains no rods, but only cones. There being many more cones per unit area here than in any other part of the retina, it follows that the fovea is the area of maximum resolution of detail; it is also the "point of fixation" which gives the observer his sense of direction in space. Monochromats usually suffer from a defect known as *nystagmus*, in which the eyes tend continually to make involuntary oscillatory movements; if monochromatism is due to "cone-blindness", the effect would naturally tend to occur by reason of the fact that the observer's eye has no point of fixation.

It is indeed fortunate that defective colour vision, especially of the more serious types, is a comparative rarity. Nature has so arranged it that human beings depend upon colour vision for many practical needs as well as for emotional pleasures. We judge the ripeness of an apple or a banana by its colour; colour often tells us whether or not a food is fit to eat, or is properly cooked. Even our bodily health is judged by this criterion; we are sometimes said to be "off-colour", and the emotion which causes us to blush would not be discernible by a dichromat. On the emotional side, the effect which colour has upon mental well-being is well known (although certainly outside the scope of this paper) and, of course, the world would be much the poorer were it never to have experienced the sensations arising from looking at a Turner or a Titian—or at a herbaceous border in midsummer.

Notes

Extraordinary General Meeting

At an Extraordinary General Meeting of the Society, held at the Victoria Hotel, Bradford, on Wednesday, 16th January 1957, at 2.20 p.m., and attended by some 38 members, it was unanimously resolved to adopt the recommendation of Council that the following addition to the bye-laws be made—

The income and property of the Society, whosoever derived, shall be applied solely towards the promotion of the objects of the Society and no portion thereof shall be used for making payment of any dividend gift division or bonus in money between any of its members.

Adoption of this bye-law will enable the Society to apply for registration by the Registrar of Friendly Societies under the Scientific Societies Act 1843, which will enable it to claim such privileges as relief from local rates.

Immediately preceding the meeting, Past Presidents, Members of Council, and Chairmen and Honorary Secretaries of Committees were guests of the President (Mr. Clifford Paine) at lunch.

Meetings of Council and Committees January

Council—16th
Finance and General Purposes—16th
Publications—22nd
Colour Index Editorial Panel—11th and 29th
Fastness Tests Co-ordinating—8th
Symposium—9th
Society's Medals—7th
President-elect—9th and 16th
Review of Textile Progress—21st
Examinations Board—10th

A.S.D.C. Examination

An examination for the Associateship of the Society of Dyers and Colourists will be held on Thursday-Saturday, 30th May-1st June 1957. Completed application forms (obtainable from the Society at 19 Piccadilly, BRADFORD 1, Yorkshire) must reach the Society not later than 18th March 1957.

Deaths

We regret to report the loss by death of Mr. D. Haworth, Mr. H. Jowett, and Mr. Richard Walker (Manchester).

The Perkin Centenary Trust

The following awards for educational purposes relating to the manufacture or the application of colouring matters have been announced by the Perkin Centenary Trust—

THE PERKIN CENTENARY FELLOWSHIP is to be offered for one or two years to a graduate for the purpose of higher study. It will have a value of

£600 per annum, and will be tenable at any university, or technical college, approved by the Trustees.

THE PERKIN CENTENARY SCHOLARSHIP—Two such awards will be offered, each for two years, renewable at the discretion of the Trustees for one further year, to enable candidates employed in the appropriate industries to receive an education at a university or technical college. Each award will have a value of £300 per annum. There will be no means test for the award, and a successful candidate shall not be debarred from receiving the whole or a part of his normal salary from his employers during his tenure of the Scholarship.

PERKIN EXCHANGE LECTURESHIPS—*Short Term*—An appointment will be made by the Trustees to enable a senior teacher at any appropriate university, or technical college or other institution, to visit some similar establishment overseas in order to deliver a short course of lectures. In return, a similar visit to a British institution by some overseas scientist will be arranged.

Longer Term—A second appointment contemplated will permit the exchange of lecturers for a period of up to one year between some British university, technical college, or other institution, and a similar institution overseas.

The Perkin Centenary Trust was established in January 1956 to administer the funds subscribed to commemorate the discovery of Mauve by Sir William Perkin in 1856. The Trustees are Mr. H. Jackson (Chairman), Dr. D. W. Hill, Dr. L. H. Lampitt, Mr. W. Mathers, Mr. M. W. Perrin, and Sir Robert Robinson. The Secretary to the Trustees is Mr. J. R. Ruck Keene, to whom inquiries relating to the above awards should be addressed, c/o The Chemical Society, Burlington House, London W.1.

Worshipful Company of Dyers

Mr. Henry B. Sissmore has been elected Prime Warden, and Lt.-Col. A. R. Marshall has been elected Renter Warden.

The Worshipful Company of Feltmakers

At a meeting of the Court of the Company the following have been elected officers—

Master

Lt. Col. John Aylmer Christie-Miller, O.B.E., J.P.

Upper Warden

Mr. Thomas Collyer Summers, M.B., B.S., F.R.C.S.

Renter Warden

Sir Heneage Ogilvie, K.B.E., M.C., F.R.C.S.

Third Warden

Mr. Philip Keens

Fourth Warden

Mr. John Frederick Roy Mitchell

Review of Textile Progress

The Joint Committee of the Textile Institute and the Society responsible for the preparation of the *Review of Textile Progress* wishes to draw attention to the position regarding early volumes. A very few copies of Volume I are still available, but Volumes II and III are out of print and the type for these has been distributed. A few copies of Volumes IV-VI are still available. Owing to increased cost of storage of type the Committee has recommended that this should be held only for a limited period, and reprints are unlikely unless there is a very considerable demand.

The complete set to date (Volumes I-VII) covers the years from 1949 to 1955 inclusive (Volume VIII for 1956 is in preparation), and thus forms a comprehensive record of postwar developments in textiles. Each volume is fully indexed. As time goes on it will become increasingly difficult to procure these early volumes, so that those who wish to complete their sets are strongly urged to place orders without delay for volumes still in print.

C.A.T.C.C. McGill Scholarship

The Canadian Association of Textile Colourists and Chemists, Quebec Section Scholarship, of an annual value of \$100, has been established in recognition of the help given to the Section by McGill University, Montreal, notably in the provision of meeting rooms. The award is made to the candidate entering the final year in the Honours Chemistry Course who receives the highest aggregate standing in the semi-final year. The first scholarship, for 1956-1957, has been awarded to Mr. S. C. Blum.

Second International Congress of Surface Activity London, 8-12th April 1957

The total number of papers to be presented is some 200, including 14 on washing. Full details are available from the Honorary Secretary of the Congress at 14 Belgrave Square, London S.W.1.

Nederlandse Vereniging voor Textiel-chemie

The Dutch society celebrated its ten years of existence by holding a congress at Enschede on Tuesday and Wednesday, 23rd and 24th October 1956. The jubilee dinner was held in the Twentse Schouwburg on the Tuesday evening, and the President of the N.V.T.C., Dr. F. B. Gribnau, gave the opening address on the Wednesday morning. He outlined the story of Perkin's discovery in 1856, and showed that the organisation of dyers and colourists in national societies began with the founding of our own Society in 1884, followed much later by the Internationaler Verein der Chemiker-Coloristen (1908) and the Association des Chimistes de l'Industrie Textile (1910). The I.V.C.C. had sections in Austria, Hungary, Italy, Poland, and Switzerland. When Mussolini was in power, the Italian section was dissolved, as Italians were not allowed to be members of an international organisation, and the Associazione Italiana di Chimica Tessile e Coloristica was formed. To

maintain some sort of international contact the International Federation of Associations of Textile Chemists and Colourists was founded in 1930 under the auspices of Professor Tagliani, with its headquarters in Basle. In 1938 it included the I.V.C.C., the A.C.I.T., the A.I.C.T.C., and the Polish and the Spanish organisations, our own Society being affiliated. A Dutch section of the I.V.C.C. was formed in 1936 with 30-50 members, and continued to meet regularly until 1943. After the Second World War the sections of the I.V.C.C. in the different countries fell apart, and an independent Dutch society was founded in Utrecht on 27th April 1946. Dr. Gribnau then outlined the activities of the N.V.T.C. since its inception.

Following the opening speech Ir. J. Lotichius (who had addressed the inaugural meeting in 1946) read a paper on *Harmonious Colour Combinations*, B. Mellbin described *The Pad-Roll System*, and Ir. J. R. H. van Nieuhuys, Director of the Vezelinstituut T.N.O. in Delft, took *A Look into the Future of the Finishing Industry*.

Verein Deutscher Färber

The V.D.F. has just formed a new local group, the Hansa group, in Hamburg, which raises the total number of groups to sixteen.

New Dyehouse at the Staatliche Textilfach- und Ingenieurschule Münchenberg (Franconia, Germany)

The opening of a new building containing laboratories, lecture theatres, and an experimental dyehouse has enabled the school to set up a Department of Textile Finishing. The courses will last five semesters (half-sessions) for "engineers for textile finishing", three semesters for "textile-finishing technicians [-techniker]", and one semester for foremen.

Organisation of Small Instrument Exhibitions

Suggestions useful for the organisers of small displays of scientific instruments will be found in *Code of Practice for the Organisation of Small Instrument Exhibitions*, available free of charge from the Scientific Instrument Manufacturers' Association, 20 Queen Anne Street, London W.1.

Manufacture of Intermediates in India

According to a statement of the Indian Ministry of Heavy Industries, quoted in *Colourage* (3, 38 (Oct. 1956)), the Government of India has decided that the National Industrial Development Corporation, a private limited company owned entirely by the Government, shall undertake the manufacture of primary intermediates required for the production of dyes. The Government is anxious to ensure that, by the end of the Second Five-year Plan, the bulk of the domestic requirements for dyes shall be produced in India from indigenous raw materials, not from imported intermediates, as is the case with the present dyemaking plants in India.

The number of plants required (one or two) and the site(s) are not yet settled. The cost of the intermediates project is expected to be Rs. 18 crores

(about £15,000,000), compared with an annual import of dyes of Rs. 15 crores (5000 tons), which is expected to increase by at least 30% by 1960. Production of intermediates independently of dyemaking plants is desirable because of their importance for other industries, e.g. drugs and plastics.

The Government's decision follows reports made by British, German, and Italian experts.

OBITUARY NOTICE

Rowland Bertie Whitehead

By the sudden death of R. B. Whitehead, M.Sc., on 23rd October 1956 at the age of 55, the Society lost a keen and willing member, the Clayton Dyestuffs Co. Ltd. a devoted employee, and his colleagues a loyal friend.

After completing his studies in the Department of Colour Chemistry and Dyeing of the University of Leeds in 1926 he was for a time a chemist with the Calico Printers Association Ltd., and later became Chief Chemist of Dominion Textiles Ltd., Canada, a position which he held until 1938, when he joined Ciba Ltd., Basle, as a Technical Liaison Officer.

Tacryl—New Swedish Textile Fibre

The electrochemical company Superfosfat-Fabrikken has prepared a new textile fibre from acetylene and ammonia, which is claimed to be cheaper than existing products. It has wool-like properties, but is lighter and more resistant than wool. It is suitable for blending with wool or viscose rayon staple for the manufacture of cloths and underclothing.

Eventually, for family and health reasons, he came to England and in 1946 was appointed to the Yorkshire staff of the then Clayton Aniline Co. Ltd. All members of the staff, as well as many business acquaintances, came to appreciate his integrity and never failing kindness.

He was keenly interested in many branches of the arts and, despite indifferent health, he was very enthusiastic about all his many activities.

During the period 1947–1949 R. B. Whitehead was an Abstractor for the *Journal*, and he regularly attended local meetings of the West Riding Section.

He is survived by a widow, a son, and a daughter.

T. A. FORSTER

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

I—PLANT; MACHINERY; BUILDINGS

Corrosion Problems in Dyeing and Bleaching

M. Trede

Tidsskr. Textiltek., 14, 79–86 (1956); Chem. Abs., 50, 16112 (10 Nov. 1956)

A description of the different types of corrosion, e.g. plane, point, slit, deformation and contact corrosions, experienced with various stainless steels used in dyeing and bleaching plant. The points which must be observed for optimum prevention of corrosion are summarised. 21 references. C.O.C.

PATENTS

Continuous Wet-treatment of Freshly Formed Regenerated Cellulose Rayon

Courtaulds

BP 762,959

Liquid Treatment of Threads on a Thread-advancing Device

Courtaulds

BP 761,803

An improved method of threading freshly spun viscose rayon immersed in hot dilute acid on a roller-type thread-advancing device is described. W.G.C.

Continuous Wet Treatment of a Plurality of Yarns

Algemene Kunstzijde Unie N.V.

BP 758,671

In order to reduce turbulence, the liquid layer in which the yarns are treated is transported in the same direction as the yarns by means of a conveying device placed under the layer. W.G.C.

Simultaneously Processing a Plurality of Yarns

Industrial Rayon Corp.

BP 761,511

A method for simultaneously processing a number of yarns is described, e.g. in the continuous spinning of viscose rayon. W.G.C.

Scouring Wool or other Loose Fibres

T.M.M. (Research)

BP 760,958

Dyeing Continuous Lengths of Fabric

A. Zanger

USP 2,724,254

Apparatus in which the fabric is drawn through a series of vats by opposed sets of bands which grip the selvedges

and maintain the fabric taut during the whole process and so prevent excessive shrinkage. C.O.C.

Treating Fabric with Liquid or Gas

Flaxall Products

BP 761,469

Apparatus allowing great increase in the amount of fabric which can be treated within a given chamber comprises two rollers mounted parallel to one another and so that they may be revolved about an axis parallel to their own axes to enable their positions to be interchanged. Two plies of fabric are fed on to the first roll, the rollers' positions are interchanged and one ply is fed from the first roller on to the second roller and one ply is drawn off. At the same time the fabric is fed from the first to the second roller another length of fabric is fed to the second roller so that it has two plies on it. The rollers are then interchanged and the process repeated as long as there is fabric to be fed into the machine. C.O.C.

Conveying Warps or Cloth during Finishing

Stoffel & Cie.

BP 759,476

Cloth Guiding Devices

John Dalglish & Sons

BP 761,851

Tension-responsive Control Apparatus for Weft-straighteners

J. D. Robertson

BP 761,830

Introducing Fabric into Tenting and Drying Machines

H. Krantz Söhne

BP 759,973

Drying Chambers and the like

Gebrüder Sucker

BP 758,911

Drying Impregnated Textiles with Hot Air

Gebrüder Sucker

BP 760,188

Controlling the Register in Moving Webs

J. F. Crossfield

BP 759,959

Shearing and other Machines treating Fabric by a Rotating Device

Sellers & Co. (Huddersfield)

BP 761,436–7

The electric motor driving the cutter, brush or abrasive roller of a shearing, brushing or abrasive machine, has its direction of rotation reversed by means of a detector

mechanism when a joint in the fabric approaches the cutter, brush or abrasive roller and is then either stopped or retracted until the joint has passed the cutter or the like.
C.O.C.

Raising Machine

Parks & Woolson Machine Co.

BP 760,553

Rollers for furnishing Anti-static Agents when Processing Textiles

Wool Industries Research Assocn.

BP 760,394

The material is brought into contact with a roller whose surface consists of a finely porous substance impregnated with a liquid anti-static agent. The anti-static liquid is fed into the interior of the roller.
C.O.C.

Hosiery Setting Machine

Etablissements M. Heliot

BP 760,447

Socks composed of synthetic fibres are placed on forms and introduced into a closed chamber where they are pre-heated by hot air, then steamed under pressure, and finally dried by hot air.
G.E.K.

Washing and Dry-cleaning Machines

Manlove, Alliott & Co.

BP 761,989

Rug Scouring Apparatus

Proctor & Schwartz

USP 2,725,583

Hat Finishing Machine

Doran Brothers

BP 760,454

Machine which ensures that the crown of the hat body is uniformly pounced.
C.O.C.

Screen Printing Machine (IX p. 74)

II—WATER AND EFFLUENTS

Influence of Synthetic Detergents on Sewage Treatment

P. N. Degens

World Congress on Surface-active Agents, 3, 1054-1058 (1954)

Control of the Effect of Synthetic Detergents on Sewage Treatment by Chemical Coagulation

S. Beaver

World Congress on Surface-active Agents, 3, 1059-1065 (1954)

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Viscosity of Solutions of Sodium Silicates as a Function of Composition and Temperature

L. Blanchard

World Congress on Surface-active Agents, 1, 316-321 (1954)

Separation of Alkyl Sulphates by Foam Fractionation

H. C. Evans and S. R. Epton

World Congress on Surface-active Agents, 1, 114-124 (1954)

Relation between Structure and Foam Performance of Dodecylbenzenesulphonates

P. L. Kooijman, H. W. Huijser, and J. J. Tjepkema

World Congress on Surface-active Agents, 1, 130-132 (1954)

Critical Solution Temperature of Monoalkyl Ethers of Polyethylene Glycols

P. Lambert and N. Chakovsky

World Congress on Surface-active Agents, 1, 43-46 (1954)

Effect of a Third Constituent on the Critical Solution Temperature in Water of Monoalkyl Ethers of Polyethylene Glycols

P. Lambert and N. Chakovsky

World Congress on Surface-active Agents, 1, 47-51 (1954)

Method of Production of Teepol

P. Donno

World Congress on Surface-active Agents, 1, 373-378 (1954)

Position of Nitrogen-bearing Amphoteric Compounds in the Detergent Field

M. McCutcheon

World Congress on Surface-active Agents, 3, 1319-1322 (1954)

The advantages of long-chain amphoteric compounds, such as substituted alanines, are discussed. Compounds of this type are soluble in acid and in alkali. They have good detergent properties, which reach a maximum at ca. pH 7, but they can be used successfully along with builders. Solutions foam well and are bacteriostatic.

In hard water they form lime soaps, but these are not gummy and are easily removed by rinsing. They are not compatible with fatty-acid soaps or anionic detergents. Their possible use as toilet bar soaps is suggested.
W.K.R.

PATENTS

Bisdecyl and Bistridecyl Esters of Sulphosuccinic Acid—Surface-active Agents

American Cyanamid

BP 760,121

Hydroxyaralkylene Ethers of Arylsulphonic Acids—Emulsifying Agents and the like

Dow Chemical Co.

BP 763,119

Compounds of formula—



(R¹ = benzene nucleus substituted only by < 4 Alk and having no or only one phenyl group attached; R² = a monovalent mononuclear aromatic hydrocarbon; M = H, alkali metal, alkaline earth metal, ammonium or subet. ammonium; m = valency of M; n = whole number) are obtained by condensing an aralkylene oxide with a hydroxyarylsulphonic acid. They are transparent, glassy solids, soluble in water and useful as emulsifying, wetting, penetrating and spreading agents.
C.O.C.

Milling Agent

Metallgesellschaft

BP 759,158

The milling properties of carboxylic acids of high mol. wt. are enhanced if they are used in fine aqueous dispersion obtained by using as emulsifying agent a surface active ester of phosphoric acid containing at least one substituted or unsubstituted hydrocarbon radical of > 5 C, e.g. good milling of a heavy coating (800 g./m.) was obtained with an aqueous emulsion of the acid secondary ester of orthophosphoric acid with oleyloctoglycol, Na salt of the secondary ester of orthophosphoric acid with lauryl tetraglycol ether, and oleic acid.
C.O.C.

Amphoteric Cationic Condensates

FBY

BP 759,855

Phenol sulphones are condensed with non-aromatic primary or secondary amines and formaldehyde to yield products which are soluble in acids and alkalies but insoluble near the neutral point. In their cationic form they quantitatively precipitate anionic compounds, e.g. tannins, dyes and sulphonated oils; they also precipitate gelatin to a greater or lesser extent. They have good fastness to light and may be used as auxiliary tanning agents, textile assistants and dispersing agents for cationic tannins.
C.O.C.

Sizes and Textile and Leather Finishing Agents from Polymeric Epoxides treated with Phosphoric Acid

DuP

BP 759,863

The products obtained by treating a polymeric epoxide copolymer with < 0.5 mole phosphoric acid per epoxy O atom in the copolymer are soluble in organic solvents and have wide uses including *inter alia* use as sizes or finishing agents for textiles and leather and in coating compositions. The copolymer should have an epoxide O content of 0.3-8.0% by wt., it should be made from 3-60% of an ethylenically unsaturated epoxymonomer and 97-40% of an ethylenically unsaturated acyclic epoxy-free monomer.
C.O.C.

Ethylene Imin Derivatives—Finishing Agents

Allied Colloids (Manufacturing) Co.

BP 760,311

Derivatives of ethyleneimine containing more than one



group ($x = 0$ or 1 ; R = H or Alk; T = H or Alk or, when $x = 1$, a carboxylic group or salt, ester or anilide thereof), readily polymerise or they readily react with compounds containing active groups such as NH₂, COOH, SH or phenolic OH or such inorganic ions as halide, cyanate, bisulphite or thiourea. Applied to wool they impart decreased tendency to felt, increased affinity for acid dyes and increased resistance to wear. Used on viscose rayon, they give increased resistance to creasing, increased wet and dry strength, high affinity for acid dyes and increased wet fastness to direct dyeings. Thus ethylene imine treated with methyl acrylate in methanol yields the methyl ester of *NN*-ethylene-β-alanine which in turn treated with ethylene diamine yields—

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**A kier-boiling
assistant**
for improved whiteness
and absorbency of
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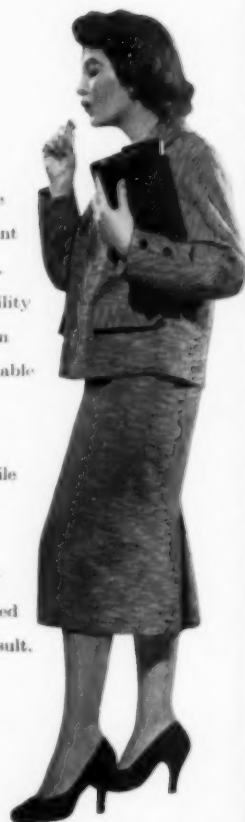
all that way in the train...

and never a single crease...


such a joy!



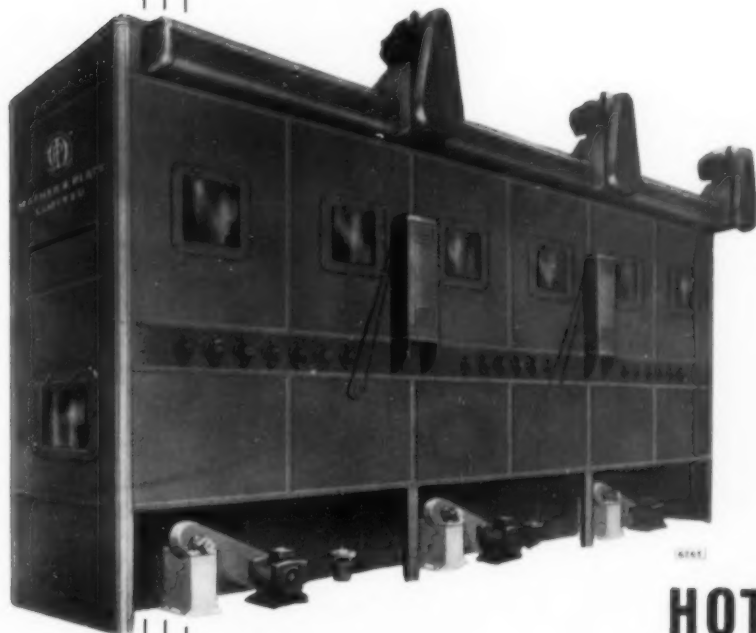
The "little extra"—which Beetle Fashion Finishes give—is a potent factor at the point of sale to-day. The buying public *expects* durability and stability of colour, dimension and effect. Therein lies an invaluable marketing opportunity for the smaller Finisher as well as the great Houses—with Beetle Textile Resins to give a wide variety of durable finishes and effects. Use them; identify the fabrics or garments as treated, and enhanced goodwill, improved sales *must* result.



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*The above machine can be seen in operation at our
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Inns and the Textile Trade



No. 8 'The Fleece', Kendal, Westmorland

By permission of William Younger & Co. Ltd.

When Shakespeare was dreaming up fat Falstaff's invective at the 'misbegotten knaves in Kendal green', The Fleece at Kendal was already well-established.

The inn is at least three centuries old, though no one seems to know its exact date. We do know, however, that the trade which gave the house its name—the sheep, the fleece, the yarn that was woven into the hardwearing cloth known as Kendal green—dates back to 1331 when Edward III gave weaver John Kemp licence to work in the town.

The trade flourished. To the Kendal

townsfolk it became all-important. So much so that blazoned with the town's coat-of-arms is the motto '*Panus mihi panis*'—wool is my bread.

The motto could apply to all of us. Wool is still one of our nation's greatest sources of revenue. The trade, though it preserves many of its ancient traditions, is constantly being studied to meet new demands and processes are carefully modernised. Some of the chemicals necessary for this important and profitable work are supplied by Brotherton.

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Target for a Boomerang



Just like a boomerang, complaints of poor colour fastness invariably return to the dyer or printer. As claims increase, business is likely to suffer. On the other hand, fast dyed goods not only please the consumer, but also ensure repeat orders. The use of Fast Dyestuffs of BASF is profitable investment.

BASF

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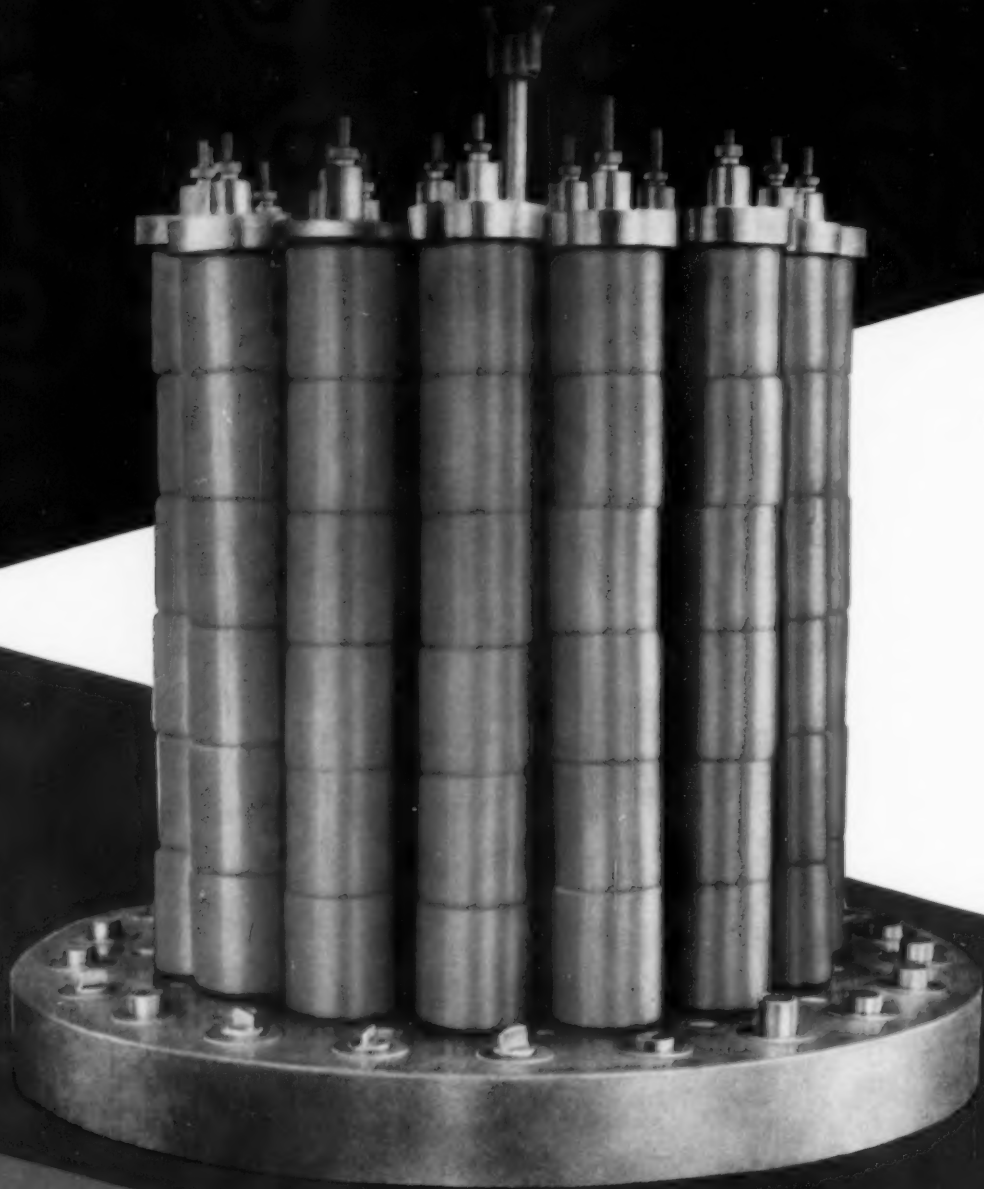
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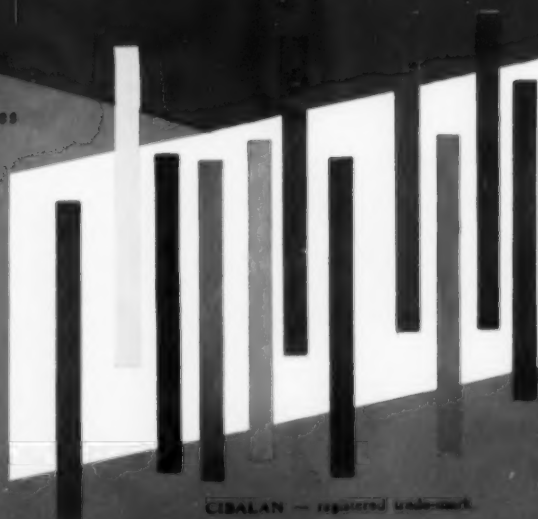
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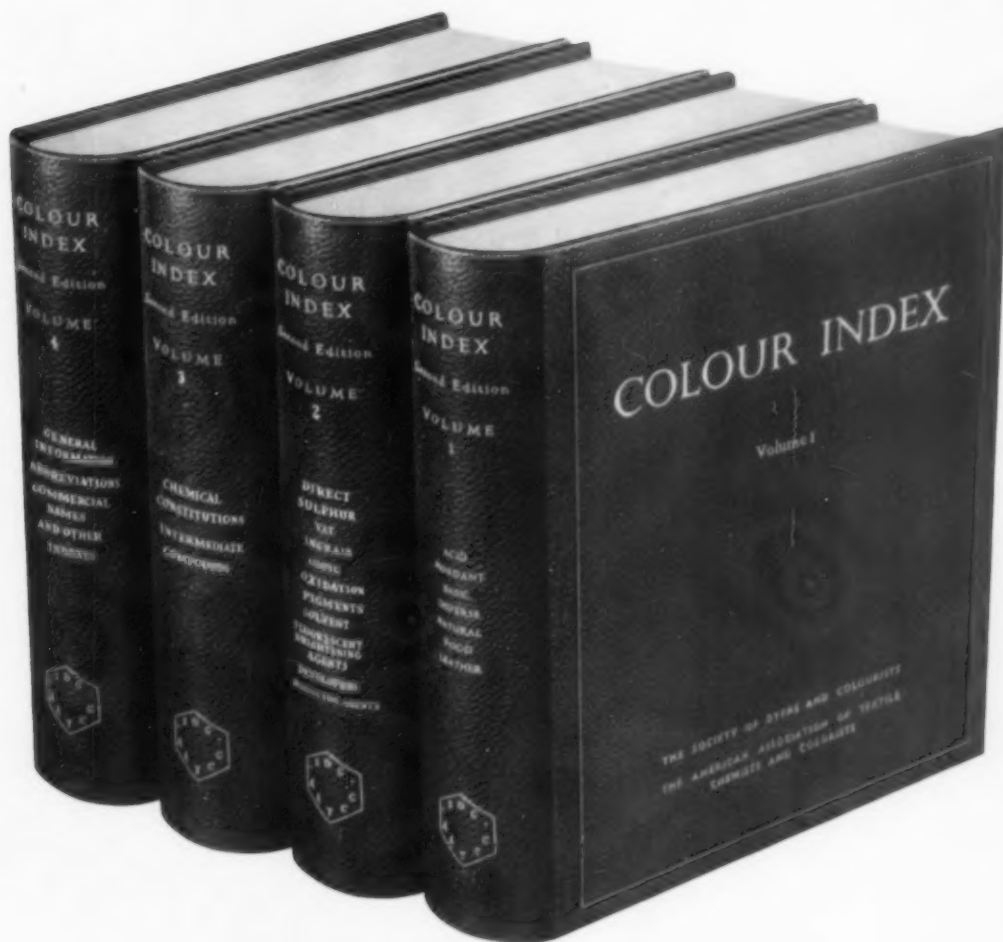
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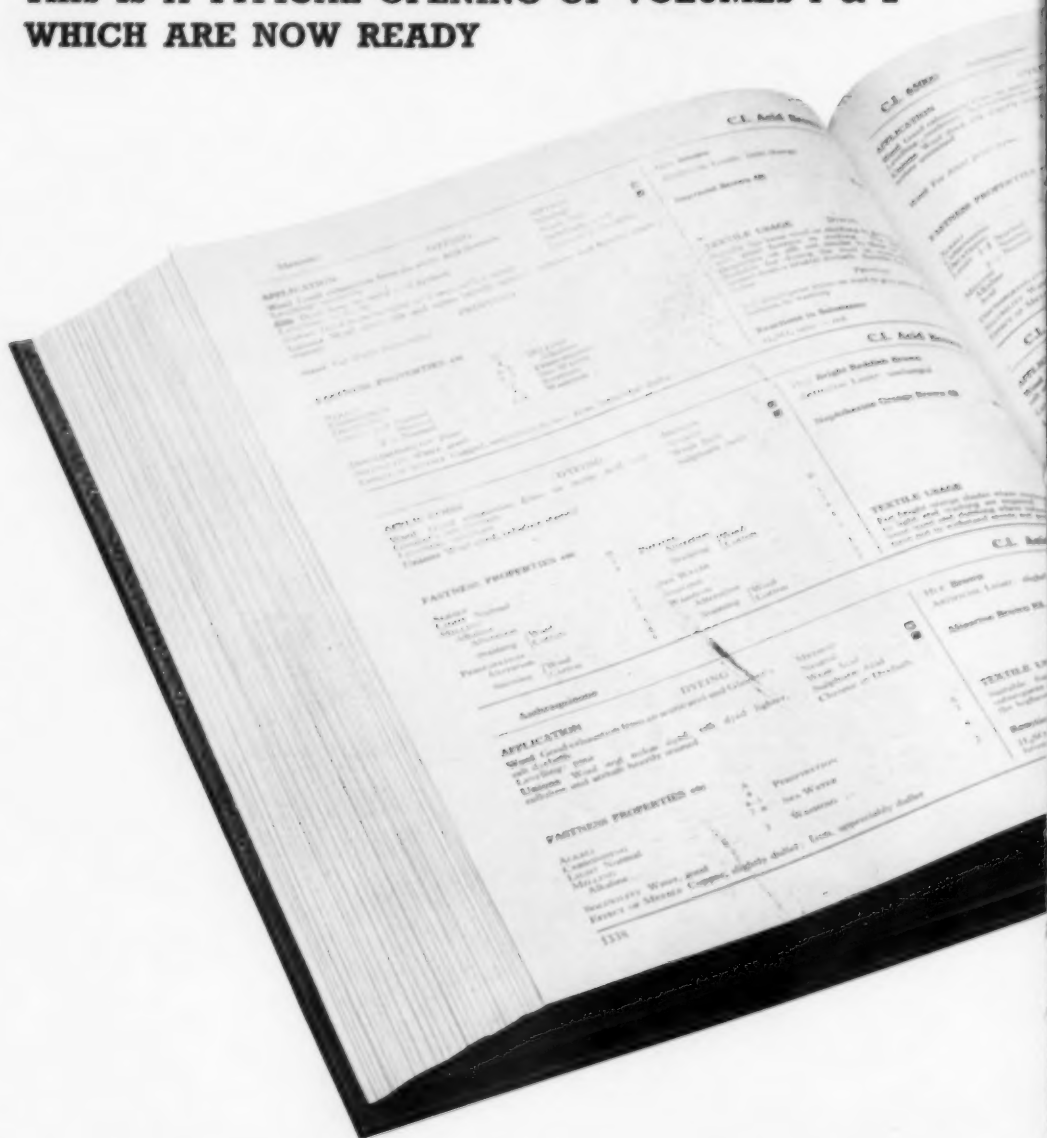
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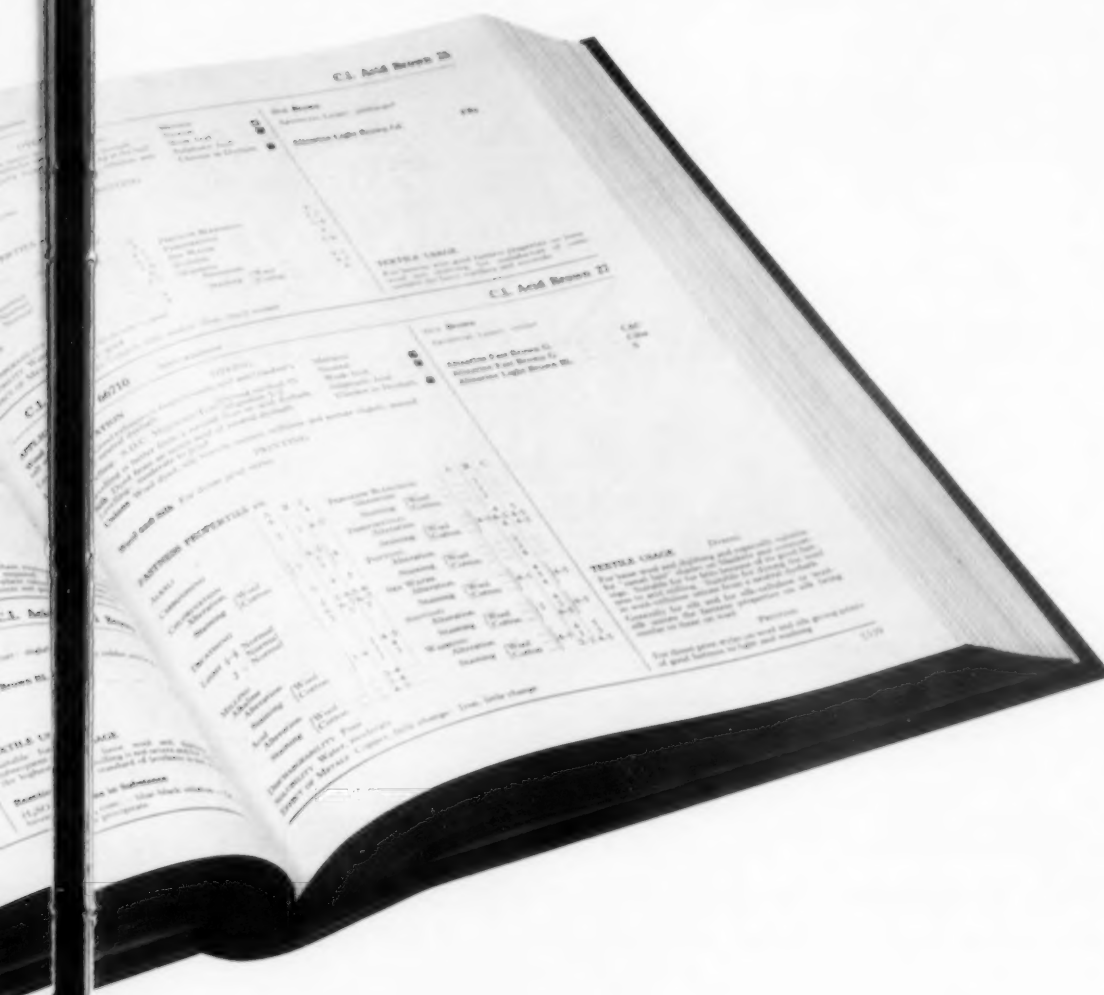
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- A — (American) The methods of The American Association of Textile Chemists and Colorists.
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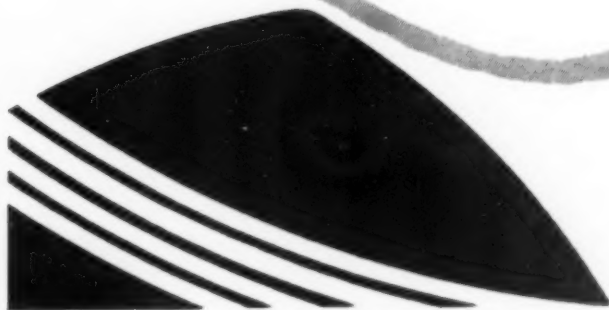
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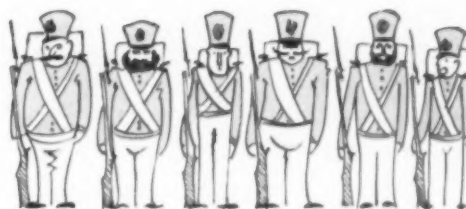
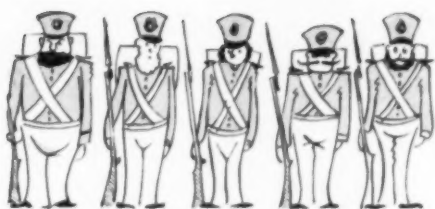
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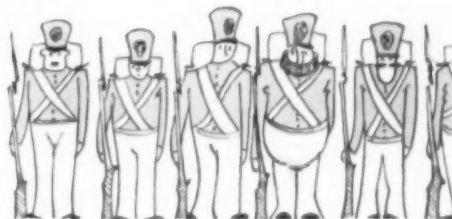
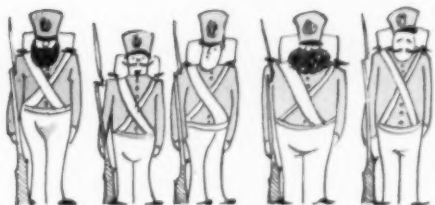
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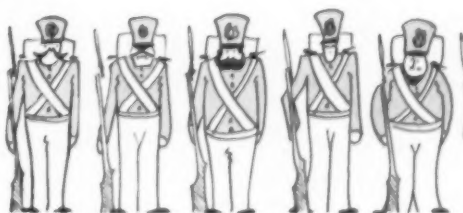
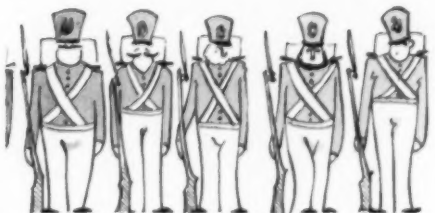
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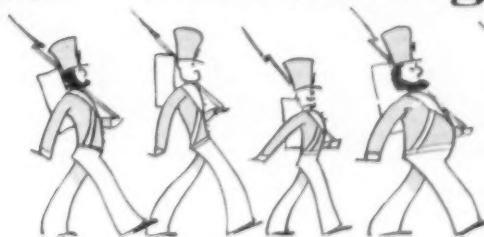
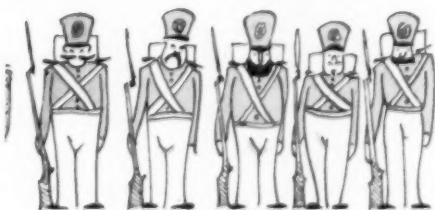
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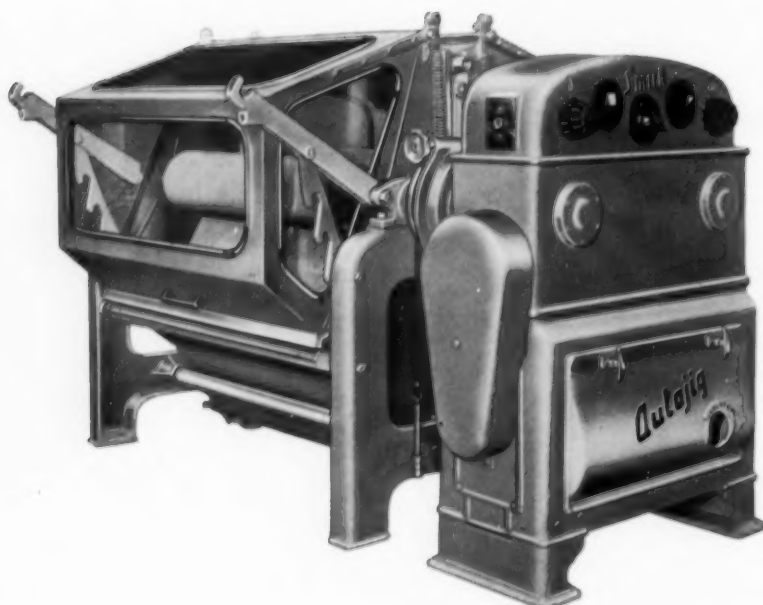
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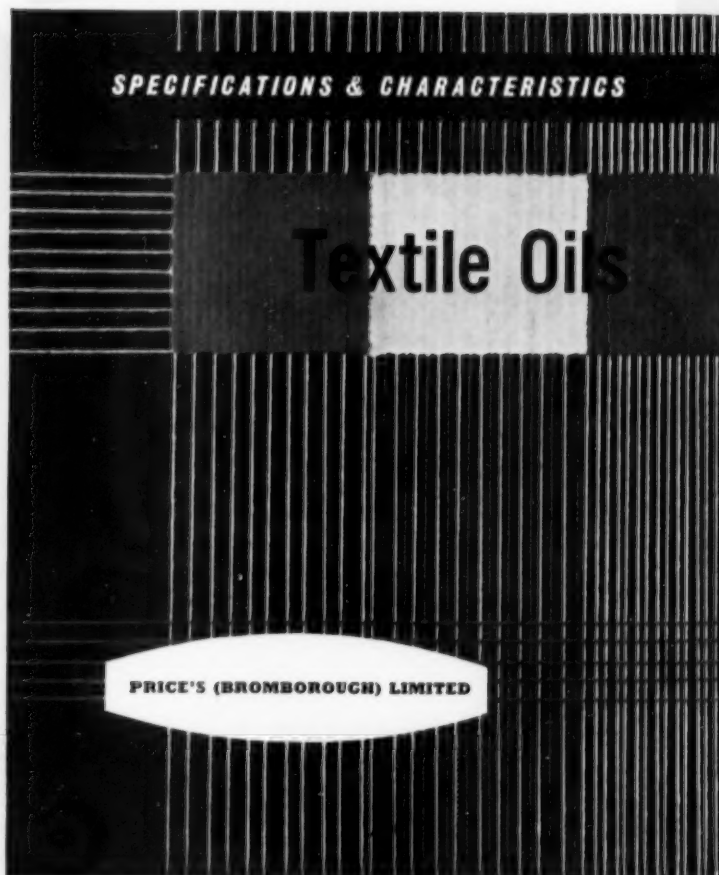
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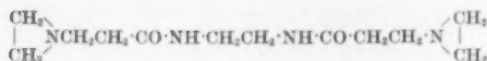
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This is very soluble in water. Textiles treated with it have increased resistance to creasing and improved dimensional stability. Material padded with a solution of this product and octadecylamine hydrochloride, dried at 75°C. and then well washed with soap and soda is water-repellent. C.O.C.

Resin Finish

Monsanto

USP 2,725,308

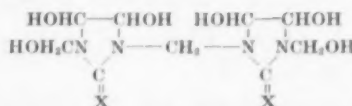
A composition comprising a water-soluble tetramethylol acetylene diurea and/or a water-soluble tetramethylol hydantoin, a water-soluble copolymer of a compound containing a single $\text{CH}_2\text{C} <$ group and an ethylenically unsaturated dicarboxylic compound (e.g. a vinyl acetate-maleic anhydride copolymer) and a water-soluble polyhydric compound containing > 1 OH group (e.g. polyvinyl alcohol) used in the normal way of applying resin finishes to cellulosic textiles yields a stiff finish which is crease-shedding and fast to washing and drycleaning. C.O.C.

Methylene-1-bis-4:5-dihydroxy-3-hydroxymethyl-imidazolidines—Resin Finishes

Prix-Werke

BP 762,344

Compounds of formula—



(X = S or O) which may be obtained in aqueous solution by reacting 1 mol. urea or thiourea with about 1.5 mol. HCHO in neutral or slightly alkaline solution at room temperature then treating with 1 mol. glyoxal, allowing to cool and stand, are used as textile finishing agents, e.g. to render rayon fabric resistant to abrasion. C.O.C.

Polymerisable Ureido Esters—Agents for Imparting Dimensional Stability to Wool Goods

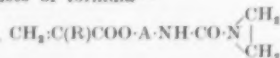
Rohm & Haas Co.

BP 761,901

An isocyanato ester of acrylic or methacrylic acid is treated with ammonia to yield products of formula $\text{CH}_2\text{C}(\text{R})\text{COO-A-NH-CO-NH}_2$ (R = H or CH_3 ; A = satd., divalent, aliphatic or cycloaliphatic hydrocarbon). These esters readily polymerise alone or with other compounds containing a $\text{CH}_2\text{C} <$ group. They are especially useful for preparing finishes to reduce the felting power of wool. Thus wool cloth impregnated with an aqueous emulsion of ethyl acrylate and β -ureidoethyl methacrylate (obtained by treating β -isocyanatoethyl methacrylate with ammonia) (955:45) and baked for 10 min. at 300°F. does not shrink when laundered.

The products of formula—

BP 762,013



obtained by treating an isocyanato ester of acrylic or methacrylic acid with ethyleneimine, have similar properties and use. C.O.C.

Quaternary Ammonium Catalysts for use with Acetylene Diurea Derivative Resin Finishes

Dan River Mills

USP 2,724,664

Compounds of formula $\text{R-A-CH}_2\text{-P-X}$ (R = aliphatic of 14-22 C; P = pyridinium or alkyl-subst. pyridinium; X = anion of a strong acid; A = amide, ester or ether group), e.g. stearamidomethyl- β -picolinium nitrate, have marked superiority over other catalysts for the reaction between acetylene diurea derivatives and neutralised cellulose. C.O.C.

Polymeric Finishing Agents

Bradford Dyers' Assn. and Albright & Wilson

BP 761,985

The products obtained by simultaneously condensing and polymerising together a tetrakis-hydroxymethyl phosphonium salt or trishydroxymethyl phosphine oxide and a nitrogenous polyfunctional compound having > 1 H atom attached to N or H linked to C on an aromatic nucleus and/or a precondensate of such a nitrogenous compound and an aldehyde, e.g. tetrakis-hydroxymethyl

phosphonium chloride, triethanolamine and methylol melamine, have varied uses as finishing agents. The finish produced depends on the fibre and the components used to produce the finishing agent. Thus cellulosic textiles may be given increased resistance to creasing and increased dimensional stability, wool textiles may be given increased dimensional stability, modified dyeing properties or the fixation of mechanical finishes. Inflammable fabrics may be rendered flameproof and resistant to afterglow. C.O.C.

Flame-resisting Finishing Agents

FBy

BP 762,169

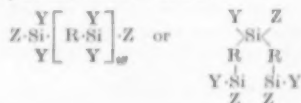
Heating 1, 2 or more mol. of dicyanamide with boric acid at 200-500°C. yields products which are very sparingly soluble in water. Incorporated in a resin solution they are used for imparting a flame-resisting finish to textiles. C.O.C.

Stable Aqueous Silanol Dispersions

Libbey-Owens-Ford Glass Co.

BP 758,640

A hydrolysable silane of formula—



(R = organic radical of 1-12 C; w = 1 or 2; Y = a hydrolysable group; Z = a monovalent organic radical of 1-12 C or a hydrolysable group) e.g. methyl trichlorosilane, is added to an aqueous solution of a buffer to produce a dispersion of pH 3-8. The products are used to impart water-repellency to various materials, e.g. siliceous materials and carbohydrates including glass, ceramics and cellulose. C.O.C.

Organopolysiloxane Water-repellent Compositions

Société des Usines Chimiques Rhône-Poulenc

BP 758,987

A composition particularly suitable for imparting to fibrous organic materials a water-repellent finish which is fast to washing comprises an aqueous emulsion containing 20-61% by wt. of a methyl polysiloxane resin of CH_3Si ratio 1:3:1 to 1:7:1, 25-50% of a methyl polysiloxane oil of CH_3Si ratio 1:9:1 to 2:1:1, and 7.5-40% of triethanolamine titanate. C.O.C.

Polymerisable isoCyanato Esters

Rohm & Haas Co.

BP 759,860

Esters of formula Z-COO-A-NCO (A = satd. aliphatic or satd. cycloaliphatic or aryl substituted satd. aliphatic, divalent hydrocarbon group of < 15 C, there being at least two C atoms between the carboxy and isocyanato groups; Z = CH_2CH , $\text{CH}_2\text{C}(\text{CH}_3)$, $\text{CH}_2\text{C}(\text{Cl})$ and $\text{CH}_2\text{CH}(\text{CH}_3)$ or their polymers or copolymers with other compounds containing at least one vinylidene group are good cross-linking agents for cellulosic compounds and proteinaceous materials. In addition, they can themselves be cross-linked and so rendered insoluble and infusible. C.O.C.

Tin Catalysts for Curing Polysiloxane Resins

G. P. Mack and E. Parker

USP 2,727,875

Some water-soluble complex organotin compounds, e.g. the compounds obtained by treating hydroxy acids containing at least one alcoholic hydroxyl group and at least one carboxyl group with stannous hydroxide, are excellent catalysts for the polymerisation of polysiloxane resins. They are particularly suitable when such resins are used together with water-soluble aminoplast resins to impart a crease-shedding and water-repellent finish to textiles. C.O.C.

Water-soluble Chromium Complexes—Water-repellent Agents

Fran.

BP 761,834

An alkali metal chromate, a carboxylic acid of > 3 C, and alcohol or aldehyde and a gaseous hydrogen halide are reacted together to yield a water-soluble complex salt of chromium and the acid. They are used as water-repellent agents and for coating glass and plastics. Thus a mixture of stearic acid, Na bichromate and alcohol in CCl_4 is refluxed while dry HCl is passed through it until the HCl is no longer absorbed. Refluxing is continued for another 30 min. and then the solvent evaporated. The resulting mass is taken up in boiling isopropyl alcohol, cooled and filtered. Fabric is impregnated with an equal weight of

this solution and of an aqueous solution containing urea, Na formate and HCOOH and dried for 5 min. at 120°C. An excellent water-repellent finish is imparted which is fast to washing. C.O.C.

Stabilised Organic Titanium and Zirconium Compounds

National Lead Co. BP 763,010

Solutions of aminoalcohol esters of titanate or zirconate acid are stabilised by adding a polyhydroxy aliphatic or alicyclic alcohol or a monobasic hydroxy acid. C.O.C.

Flame-resistant Finish for Cellulosic Materials (IX p. 74)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Light Absorption Studies

V—Relation of Mesomeric Effects and Ultra-violet Absorption Spectra

W. F. Forbes and A. S. Ralph

Canadian J. Chem., **34**, 1447-1456 (Oct. 1956)

VI—Effective Interference Values of Non-bonded Atoms in Solution: I—Some Preliminary Observations

W. F. Forbes and W. A. Mueller

Ibid., **34**, 1542-1552 (Nov. 1956)

Spectroscopic Interaction between Unconjugated Chromophores. Intramolecular Charge-transfer Spectra

R. C. Cookson and N. Lewin

Chem. and Ind., 984-985 (22 Sept. 1956)

Studies in Peroxidase Action. XI—Oxidation of a Mixture of Amines

G. M. K. Hughes and B. C. Saunders

J.C.S., 3814-3820 (Oct. 1956)

The peroxidase oxidation of aniline and *p*-toluidine together is now investigated to determine whether or not the separate products of the single oxidations are formed. It is found that, if the mixed oxidation proceeds too far, tarry products of high mol. wt. are formed which arise from simpler intermediates. When the oxidation is allowed to proceed to only 20% completion, 4,4'-dimethylazobenzene (I) may be isolated together with a dimethyl deriv. of azophenine, a probable homologue of 2-hydroxy-5-anilino-*p*-benzoquinone dianil, and a probable mono-*p*-methyl analogue of 2-amino-5-anilino-*p*-benzoquinone. Apart from I, none of the products resulting from each amine alone has been obtained from the mixture (cf. *J.C.S.*, 940 (1956)). H.H.H.

Aromatic Substitution. II—Nitration of Aromatic Compounds with Nitronium Tetrafluoroborate and other Stable Nitronium Salts

G. Oláh, S. Kuhn, and A. Minkó

J.C.S., 4257-4258 (Nov. 1956)

A new method of nitration of aromatic cpd. is reported based on the use of stable nitronium salts, viz. the tetrafluoroborate, hexafluoroarsenate, hexafluoroantimonate, and hexafluorophosphate. Of these the tetrafluoroborate, $\text{NO}_2^+ \text{BF}_4^-$, is the most convenient, and can be prepared from nitryl fluoride and boron trifluoride or by the reaction of dinitrogen pentoxide with hydrogen fluoride and boron trifluoride in nitromethane; it is a white cryst. cpd., stable in the absence of moisture and adaptable as a nitrating agent for common use; its salt-like character has been proved by electrical conductivity measurements. This new method gives a direct preparative proof of the electrophilic character of nitration through the NO_2^+ cation. H.H.H.

Mixed 2-Methylnaphthols as Dye Intermediates

F. J. Batung

Univ. Microfilms (Ann. Arbor, Mich.) Publ. No. 15612, 77 pp. (microfilm, \$1; paper enlargement \$7.70);

Dissertation Abstr., **16**, 1236 (1956);

Chem. Abs., **50**, 14673 (25 Oct. 1956)

Colour Couplers for Subtractive Colour Photography

N. Kunimine

J. Soc. Sci. Phot. Japan, **17**, 121-9 (1955);

Chem. Abs., **50**, 15297 (10 Nov. 1956)

Several 1-phenyl-3-arylsulphonamido-5-pyrazolones were synthesised by adding 2 mol. sulphonyl chlorides of aryl or heterocyclic ring compounds to 1 mol. 1-phenyl-3-

amino-5-pyrazolone in benzene, dioxane or pyridine. Comparison of the dye images shows that the 3-sulphonamido compounds have higher coupling activity than the corresponding acrylamino derivatives. The absorption of the 3-sulphonamidopyrazolone azomethine dyes, either in methanol or in dye images, compares favourably with that of the 3-acrylamidopyrazolone azomethine dyes. C.O.C.

Naphthalene Series

XVIII—Preparation of Aroyl-1-naphthols and Use of Acyl- and Aroyl-1-naphthols as Coupling Components

R. D. Desai and R. M. Desai

J. Sci. Ind. Research (India), **14 B**, 498-504 (1955)

A series of 52 azo dyes were prepared from α -naphthols substituted in the 2- or 4-position by CH_3CO , $\text{C}_2\text{H}_5\text{CO}$, $\text{C}_3\text{H}_7\text{CO}$, $\text{C}_6\text{H}_5\text{CH}_2\text{CO}$, Bz and 2- or 4- $\text{CH}_3\text{OC}_6\text{H}_4\text{CO}$ by coupling with diazotised 2-amino-4-chloroanisole, *o*- and *m*-chloroaniline, or diaminidine. Introduction of ketonic groups has marked effect on the hue, fastness and affinity of the dye for cellulose. Most were light red and had poor fastness to light but good fastness to soaping, rubbing and bleaching.

XIX—Preparation and Use of the Hydroxy Ketones of β -Naphthol as Coupling Components

Ibid., 505-8

The 6-acyl- β -naphthol dyes prepared by coupling the above amines with β -naphthol substituted by CH_3CO , $\text{C}_2\text{H}_5\text{CO}$ and $\text{C}_3\text{H}_7\text{CO}$ in 6-position are brighter and more deeply coloured than the above α -naphthol dyes but they have less affinity for cellulose.

XX—Preparation and Use of Arylamides of Schaeffer's Acid as Coupling Components

Ibid., 509-512;

Chem. Abs., **50**, 14672-3 (25 Oct. 1956)

The anilide, *p*-toluidide, α - and β -naphthylamide and benzidide of 2-naphthol-6-sulphonic acid were prepared and coupled with the above amines. The dyes produced were fast to soaping and rubbing and moderately fast to bleaching and light except those from the benzidide which have poor fastness to bleaching and soaping. They yield even and bright but rather light dyeings. They have less affinity for cellulose than the above ketones of α -naphthol.

Physical and chemical properties of all the dyes mentioned in the three parts are given. C.O.C.

Derivatives of 2:3-Dihydroxynaphthalene

F. H. Howell and D. A. H. Taylor

J.C.S., 4252-4256 (Nov. 1956)

Steric Effects in Electronic Spectra

J. N. Murrell

J.C.S., 3779-3784 (Oct. 1956)

The importance of steric effects in determining the electronic spectrum of a composite mol. is related to the interaction between locally excited and electron-transfer states of the separated fragments. The intensity of a transition to a typical electron-transfer state is in general more sensitive to steric factors than a transition to a typical locally excited state, but the latter is usually more sensitive in its frequency. The electronic spectra of the diphenyls, dimethylanilines, acetophenones, and cyanine dyes are then briefly discussed, and the nature of their electronic bands is determined from the effect of *ortho* substituents on their spectra. H.H.H.

Synthetic and Oxidative Studies in the Polyhydroxy-diphenyl Series

J. M. Bruce and F. K. Sutcliffe

II—5:6-Dichloro-2:3-dihydroxydiphenyl

J.C.S., 3820-3823 (Oct. 1956)

Nitration of 4:5-dichloroveratrole gives the 3-nitro-deriv., from which reduction and subsequent diazotisation followed by treatment with KI affords 4:5-dichloro-3-iodoveratrole. The latter when mixed with iodobenzene and treated at 225°C. with activated copper bronze affords both 5:6-dichloro-2:3-dimethoxy-(I) and 5:6:5':6'-tetra-chloro-2:3:2':3'-tetramethoxy-diphenyl. I, however, is more conveniently prepared by treatment of 2:3-dimethoxydiphenyl with sulphuryl chloride; it is readily demethylated, and the resulting dihydric phenol may be converted by mild oxidants (Ag_2O in dry ether, aq. sodium iodate, etc.) into the corresponding diphen-quinone. H.H.H.

III—2:3-Dihydroxy-5:6-dimethyldiphenyl

Ibid., 3824–3829
4:5-Dimethylveratrole is produced in 80% yield by a three-stage reaction from veratraldehyde, and also by reaction of 4:5-dichloroveratrole with methyl-lithium. With ethereal *n*-butyl-lithium it gives 13% of the 3-lithio deriv., which with CO₂ affords the corresponding carboxylic acid; the expected *tert.* alcohol was not obtained with cyclohexanone. Ethereal 3-bromo-4:5-dimethylveratrole (I) is largely debrominated by lithium or phenyllithium. 2:3-Dimethoxy-5:6-dimethyldiphenyl is formed in low yield by the crossed Ullmann reaction between I and iodobenzene; it is readily demethylated, and the resulting dihydric phenol is converted by mild oxidants into the corresponding diphenquinone. H.H.H.

Intramolecular Rearrangement of Phenylnitramine and the Benzidine and Semidine Changes

S. Brownstein, C. A. Burton, and E. D. Hughes

Chem. and Ind., 981 (22 Sept. 1956)

An isotopic investigation of the acid-catalysed rearrangements of phenylnitramine is conducted in 74% H₂SO₄ at –20°C. in the presence of excess of enriched ¹⁵N tracer as labelled KNO₃. The *o*- and *p*-nitroanilines formed are isolated chromatographically, and their ¹⁵N content determined by conversion of total N to NH₃. The sensitivity of the measurements are such that, if 1% of the rearrangement occurred by the intermolecular mechanism, it could be easily detected. Both isomers, however, are isotopically normal in N, and it is, therefore, established that *o*- and *p*-nitroanilines are formed by intramolecular processes. The results are interpreted by a scheme which has the merit that, apart from the nitro-nitrito transformations previously discussed (cf. Hughes and Ingold, *Quart. Rev. Chem. Soc.*, **6**, 34 (1952)), six-membered cyclic transition states are involved, both in the *ortho* change and in the leap-frog sequence giving the *para* isomer. The benzidine and semidine changes are interpreted in analogous manner. Schematic illustrations are included. H.H.H.

Benzimidazoles. VI—Azo Dyes

F. Montanari

Gazz. chim. ital., **85**, 981–8 (1955);

Chem. Abs., **50**, 16111 (10 Nov. 1956)

Azo dyes were made by coupling dimethyl- and diethylaniline and 1- and 2-aminonaphthalene, with diazotised 5-aminobenzimidazoles and their use for dyeing nylon and cellulose acetate tested. C.O.C.

Decomposition of Arylazotriarylmethanes

G. L. Davies, D. H. Hey, and G. H. Williams

J.C.S., 4397–4408 (Nov. 1956)

The energies and entropies of activation are measured for the decomp. of phenylazotriphenylmethane in toluene, nitromethane, nitrobenzene, acetic acid, and *n*-propyl alcohol, and of a number of subst. arylazotriarylmethanes (*p*-X-C₆H₄-N=N-C(C₆H₅)₃, where X = CH₃, Cl, Br, and NO₂) in toluene soln. Although only small differences in the rates of decomp. are shown, the activation parameters exhibit much more pronounced variations, which tend to compensate one another. The mechanism of the decomp. of these and allied cpd. is discussed, and it appears that Wieland's mechanism (cf. *Annalen*, **514**, 145 (1934)) involving primary dissociation is consistent with the known facts concerning the decomp. of azo cpd. of this type. H.H.H.

Theoretical Investigation of the Chemical Reactivity of Benzimidazole

R. D. Brown and M. L. Heffernan

J.C.S., 4288–4291 (Nov. 1956)

Bisquaternary Ammonium Salts. II—Salts of 4:4'-Diaminostilbene-2:2'-disulphonic Acid

W. G. Leeds and R. Slack

J.C.S., 3941–3944 (Oct. 1956)

4:4'-Diaminostilbene-2:2'-disulphonic acid is found to form sparingly water-sol., highly cryst. salts with a wide variety of bisquaternary ammonium bases (12 examples). These salts provide convenient intermediates for the conversion of one sol. salt into another, since they are quant. decomposed by treatment with appropriate mineral or org. acids. H.H.H.

Synthesis of Aliphatic-Aromatic Unsaturated Alcohols—Analogues of Triphenylcarbinol. II—Aliphatic-aromatic Analogues of Triphenylmethane Dyes

B. N. Dashkevich and I. V. Smolanka

Ukrain. Khim. Zhur., **21**, 619–624 (1955);

Chem. Abs., **50**, 15486 (10 Nov. 1956)

Two new ketones were made: 1-phenyl-5-(*p*-dimethylaminophenyl)-1:4-pentadien-3-one (I) from BzH and *p*-(CH₃)₂NC₆H₄CH:CHOCH₃ with NaOH, and 2-methyl-6-(*p*-dimethylaminophenyl)-2:5-hexadien-4-one (II) from *p*-(CH₃)₂NC₆H₄CHO and mesityl oxide in NaOH. I treated with C₆H₅N(CH₃)₂ and POCl₃ yielded 1:3-di-(*p*-dimethylaminophenyl)-5-phenyl-1:4-pentadien-3-ol (III) which dyes cotton bluish bordeaux. I and (C₆H₅)₂NH with POCl₃ give 1-phenyl-5-(*p*-dimethylaminophenyl)-3-(*p*-phenylaminophenyl)-1:4-pentadien-3-ol which dyes cotton greyish blue, or bluish green on tannin mordant. 2-Methyl-4:6-di-(*p*-dimethylaminophenyl)-2:5-hexadien-4-ol from II, C₆H₅N(CH₃)₂ and POCl₃ dyes tannin mordanted cotton dark violet. C.O.C.

Naphthol AS

G. Modena

Ricerca sci., **26**, 1451–1464 (1956);

Chem. Abs., **50**, 16111 (10 Nov. 1956)

Substantivity (–*A*₉₀) of a dye is defined as thermodynamic affinity; it is measured by uptake of dye when 1 g. of cotton yarn (to a precise specification) is treated in a 20 cc. dyebath for 12 hr. at 25°C., extracted with solvent, 240–260 mμ absorption of the extract (dil 1:10⁶ with 0.1 *N*-NaOH) measured with a Beckman DV spectrophotometer to determine the concentration and subtracting it from the original concentration similarly determined. C₁₀H₆(OH)CONHC₆H₄X (I) (X = H, *m*- and *p*-Alk, Hal, etc.) were prepared and properties ascertained. All *meta* substituents cause the same increase of substantivity of I; the *p*-X cause the greater increase except CH₃, OCH₃ and N(CH₃)₂. C.O.C.

Upgrading of Extracts from the Refining of Lubricating Oils with Furfural. Conversion of the Extract of Fraction V to Organic Intermediates and Dyes—I

I. Matei, E. Cocca, and C. Mărculescu

Acad. rep. populare Române, Filiala Iasi, Studii

ceretari stiint., **4** (1–4), 267–271 (1953);

Chem. Abs., **50**, 15056 (25 Oct. 1956)

The extract of fraction V was sulphonated and after removal of paraffin wax by naphtha, a black water-soluble product was obtained. Treatment with HNO₃ (d. 1.4) or dil. HNO₃ and conc. H₂SO₄ yielded materials which dyed wool chestnut to reddish chestnut. C.O.C.

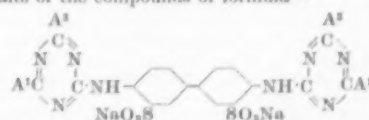
Fluorescent Brightening Agents of the Benzidine Series. III—Synthesis of Benzidine-3:3'-disulphonic Acid Derivatives containing a Triazine Ring

K. Inukai and Y. Maki

Repts. Govt. Ind. Research Inst. Nagoya, **2**, 207–211 (1953);

Chem. Abs., **50**, 16111 (10 Nov. 1956)

Na salts of the compounds of formula—



were made and of them NN'-bis(2-ethoxy-4-amino-1:3:5-triazine-6-yl)benzidine-3:3'-disulphonic acid and NN'-bis(2:4-diamino-1:3:5-triazin-6-yl)benzidine-3:3'-disulphonic acid had the strongest fluorescence.

IV—Fluorescence and Dyeing Properties of Various Compounds

K. Inukai and T. Veda

Ibid., 267–271

The fluorescence, intensity, fastness to light, affinity for fibres, etc. of the benzidine fluorescent brightening agents described in the three previous papers are tabulated and discussed. C.O.C.

Technology of Modern Oxidation Hair Dyes—II

G. S. Kass

Am. Perfumer Aromat., **68** (2), 35–7 (1956);

Chem. Abs., **50**, 15027 (25 Oct. 1956)

The oxidation bases mainly used are *p*- and *o*-benzenediamines and aminophenols and derivatives. Hue and

depth of dyeing are controlled by the concentration of the bases, the solvents, alkalinity and development time. *p*-Phenylene- and *p*-tolylenediamine are the foundation bases for dark dyeings and for many light dyeings. Blonde and red dyeings are mainly founded on aminophenols and *p*-aminophenol respectively. Other essential components of the compositions are colour modifiers and stabilisers. Antioxidants, e.g. Na_2SO_3 , are also often used. 35 references. C.O.C.

Some Recent Developments in the Chemistry of Photographic Sensitising Dyes

L. G. S. Brooker

14th Congr. intern. chim. pure et appl., Zurich, 1955; *Experientia*, Suppl. No. 2, 229-257 (1955)

Economical Advantages obtained in preparing 1-Methylamino-4-*p*-toluidinoanthraquinone

G. M. Reiner

Chimica (Milan), 12, 98-102 (1956); *Chem. Abs.*, 50, 15487 (10 Nov. 1956)

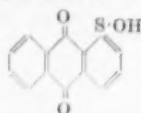
A new and cheaper way to make the 1-methylamino-4-*p*-toluidinoanthraquinone, used to dye mineral oil blue, is to sulphonate anthraquinone with oleum at 100-150°C. in presence of HgO , and treat the mass with KCl to obtain the *K*- α anthraquinone sulphate. This is treated for 12 hr. in an autoclave at 132-135°C. with NaClO_2 , CH_3NH_2 and water to give 1-methylaminoanthraquinone which is dissolved in nitrobenzene at 90°C., cooled to 25°C. and treated with Br_2 to yield 1-methylamino-4-bromoanthraquinone. This is fused with *p*-toluidine at 60-65°C. and Na acetate and $\text{Cu}(\text{CH}_3\text{COO})_2$ added, raised to 130°C. in 2 hr. and kept at 160°C. for 4 hr. After cooling the mixture is poured into dil. HCl , the dye filtered off and purified by dissolving in H_2SO_4 and then precipitating in water. C.O.C.

Organic Sulphur Compounds. I—Structure of Anthraquinone-1-sulphenic Acid, its Derivatives and Analogues

J. A. Bartrop and K. J. Morgan

J.C.S., 4245-4251 (Nov. 1956)

The structures of the title substance, its potassium salt, and its methyl ester are discussed from chemical evidence and from an examination of their infrared and ultraviolet spectra. The structure below is suggested as the most probable one for the acid—



H.H.H.

3:4-5:6-10:11-12:13-Tetrabenzoperopyrene

E. Clar and W. Kelly

J.C.S., 3875-3877 (Oct. 1956)

The title cpd. (I) is synthesised starting from 1:5-dichloroanthraquinone and 2-methyl-1-naphthylmagnesium bromide. It crystallises from trichlorobenzene in orange feathery needles, m.p. 487-488°C., the soln. having a strong green fluorescence, and it dissolves in hot conc. H_2SO_4 to give a green soln. I is different from Scholl and Meyer's dinaphthocarone, which is probably a dinaphthopyrene, and also is not identical with a hydrocarbon, claimed to be tetrabenzoperopyrene, obtained from the hydrogenation of coal.



(I)

H.H.H.

Circumanthracene and Dinaphtho(7':1'-1:13)(1'':7''-6:18)peropyrene

E. Clar, W. Kelly, J. M. Robertson, and M. G. Rossmann

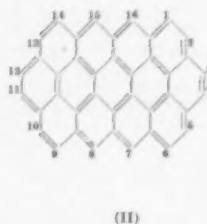
J.C.S., 3878-3881 (Oct. 1956)

Dipernaphthyleneanthracene when condensed doubly

with maleic anhydride gives a cpd. which, when decarboxylated with copper powder and quinoline, affords the orange-red dinaphthoperopyrene (I), whose absorption spectrum is reproduced. Decarboxylation with soda-lime yields I together with a more condensed hydrocarbon (II), whose structure is proved by X-ray examination. The properties of II indicate that its formation may be due to controlled graphitisation, and it is named *circumanthracene*, because of the central anthracene complex being completely surrounded by condensed benzene rings.



(I)



(II)

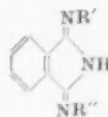
H.H.H.

Compounds containing Directly Linked Pyrrole Rings. II—Dialkylimino- β -isoidindigo

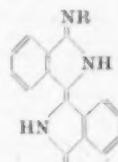
J. A. Elvidge and J. H. Golden

J.C.S., 4144-4150 (Nov. 1956)

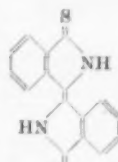
1:3-Dimethyliminoisoidindole (I: $\text{R}' = \text{R}'' = \text{CH}_3$) reacts with methylamine at 200°C. to give dimethylimino- β -isoidindigo (di-(3-methylimino-1-isoidindolylidene)) (II: $\text{R} = \text{CH}_3$), which on oxidative hydrolysis gives 2 mol. of phthalimide. Dibutyl- and dibenzyl-imino- β -isoidindigo (II: $\text{R} = \text{C}_4\text{H}_9$ and $\text{CH}_2\text{C}_6\text{H}_5$) are obtained analogously; they can also be prepared from dithio- β -isoidindigo (III) with butylamine and benzylamine respectively. Condensation of morpholine with dithio- β -isoidindigo gives a deriv. with the fixed-bond structure (IV). Light absorptions are given, and the fine structure of the new β -isoidindigo deriv. is discussed.



(I)



(II)



(III)



(IV)

H.H.H.

Vibrational Frequency Correlations in Heterocyclic Molecules. II—Infrared Spectra and Structure of Oxindole and Related Compounds

A. E. Kellie, D. G. O'Sullivan, and P. W. Sadler

J.C.S., 3809-3813 (Oct. 1956)

To supplement previous work on subst. isatins and to provide further basic information on structures containing NH and CO groups in five-membered rings, infrared spectra have been obtained of oxindole, phthalimidine, phthalimide, and related mol. in CHCl_3 . Strong support has thereby been given for fully ketonic formulae in all these cpd. Internol. H-bonding is also present provided that the NH group remains unsubst. The carbonyl

stretching frequencies of oxindole vary with the substituents in the benzene ring, probably being dependent on their σ -values. The effect of these substituents is transmitted through the NH to the CO group. Other features of these spectra are discussed in relation to the spectra of subst. isatin. H.H.H.

Effects of X-Rays on Dilute Aqueous Solutions of Amino Acids [Formation of Melanin]

J. Nosworthy and C. B. Allsopp

J. Colloid Sci., **11**, 565-574 (Oct. 1956)

The effects of X-rays on aqueous solutions of β -phenylalanine, tyrosine, and 3:4-dihydroxyphenylalanine (dopa) in the presence of oxygen have been studied. In addition to oxidative deamination, reactions occur in which dopa is formed from tyrosine and an indole derivative from dopa. It is possible that irradiation of aqueous solutions of tyrosine may lead to similar oxidative changes to those occurring in the metabolic oxidation of tyrosine to a melanin pigment. W.R.M.

Colouring Matters of the Aphididae. XV—Alkaline Inversion of Erythrophenol and its Derivatives

A. W. Johnson, A. R. Todd, and J. C. Watkins

J.C.S., 4091-4093 (Nov. 1956)

Distribution of Anthocyanin Pigments in Poppies

R. M. Acheson, J. L. Harper, and I. H. McNaughton

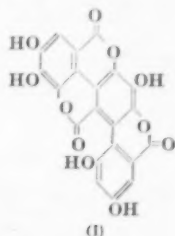
Nature, **178**, 1283-1284 (8 Dec. 1956)

Flavogallol

J. Grimshaw and R. D. Haworth

J.C.S., 4225-4232 (Nov. 1956)

The structure shown below (I), which has been considered but rejected by earlier workers, is now proposed for flavogallol on the basis of its properties and confirmation by synthesis as follows: a mixture of methyl 2-bromo-3:4:5-trimethoxybenzoate and dimethyl 5-bromo-2:3:4:2':3':4'-hexamethoxy-6:6'-diphenate is treated with Cu bronze at 220-230°C. for 2 hr., the product is demethylated by refluxing with HI and acetic anhydride, and the phenol formed is isolated as the acetate, and thence converted to flavogallol.



(I)

H.H.H.

Leguminous Dyes

G. V. Lazur'evskii

Trudy Botan. Inst. im. V.L. Komarova Akad. Nauk S.S.S.R., **5** (5), 90-119 (1955);

Chem. Abs., **50** 15743 (10 Nov. 1956)

The roots of *Sophora* species collected in Russian Central Asia contain amorphous high mol.wt. phenolic dyes to which the name "sophoreols" has been applied. These dyes are derivatives of 7-hydroxy-4-chromanone containing a partly oxidised C_{18} terpene side chain. C.O.C.

Colouring Matter of *Garcinia morella*. II—Infrared and Ultraviolet Absorption Spectra of Morellin and its Reduction Products

N. V. Bringi, M. R. Padhye, and K. Venkataraman

J. Sci. Ind. Research (India), **15B**, 128-138 (1956);

Chem. Abs., **50**, 15750 (10 Nov. 1956)

A partial structure for morellin is based on the infrared and ultraviolet spectra of morellin and a number of its reduction products. C.O.C.

Flavine Pigment of *Zelkova serrata* Wood. I

K. Nishida and K. Funaoka

Trans. 61st Meeting Japan. Forest Soc., J. Japan Forest Soc. (1952) Spec. Issue 257-8

II

Ibid., 62nd Meeting (1953), 267-9

III

K. Nishida, K. Funaoka, and M. Taraka

Trans. Kyushu Branch Japan. Forest Soc. (8), 87-8 (1953)

IV

Trans. 63rd Meeting Japan. Forest Soc., J. Japan Forest Soc. (1954), Spec. Issue 366-8

V

K. Turaoka

Ibid., 64th Meeting (1955), 337-9

A pigment has been isolated and given the name *kyakinin*. Its structure is shown to be 3:4':5-trihydroxy-7-methoxy-8-(1:2:3:4:5-pentahydroxypentyl)flavine. C.O.C.

Roumanian Attempts to prepare Mineral Pigments from Barite

M. Savul, V. Ababi, C. Braniste, and T. Idriceanu

Acad. rep. populare Române (Iasi), Studii cercetari stint., **4** (1-4), 197-212 (1953);

Chem. Abs., **50**, 15037 (25 Oct. 1956)

An account of work done in preparing $BaSO_4$ from barite, of reducing it to BaS and of the use of this in preparing lithopone. C.O.C.

Diatomaceous Calcite as an Extender Pigment for Paint

P. S. Schenk

Offic. Dig. Federation Paint & Varnish

Production Clubs, **27**, 537-550 (1955);

Chem. Abs., **50**, 16129 (10 Nov. 1956)

This natural product consists of 20% acicular diatomaceous SiO_2 and 80% of fine particle-size $CaCO_3$. It imparts to paints low sheen, good film integrity and low oil demand. Because of its low water-soluble content it is suitable for water emulsion paints. It is superior to a blend of SiO_2 and $CaCO_3$ of the same composition. C.O.C.

Vanadium Pigments

K. Sono

Repts. Govt. Ind. Research Inst., Nagoya, **2**, 63-5 (1953);

Chem. Abs., **50**, 16129 (10 Nov. 1956)

A description of preliminary experiments made to observe the colour of various V_2O_5 pigments blended with ZrO_2 , SiO_2 , SnO_2 , Al_2O_3 or CaO . C.O.C.

PATENTS

Azo Colour Couplers containing Two Coupling Nuclei

Eastman Kodak Co.

USP 2,725,291

Compounds of formula $R:N:N:R$ (R = same or different, subst. or unsubst. 4-pyrazolone, 4-phenol or 4-naphthol nucleus), e.g. 4-azobis[1-(*p*-sulphophenyl)-3-n-octyl-5-pyrazolone] are colour couplers which enable increase in the amount of dye formed in the emulsion. C.O.C.

Coloured Colour Couplers containing Solubilising Groups

Eastman Kodak Co.

USP 2,725,292

Compounds of formula—Coupler— $N:N:A-B-C$ (A = Ar ; B = an electron-releasing substituent on A ; C = a solubilising, lyophilic group), e.g. 1-phenyl-3[3-(2-(2,4-di-*tert*-amylphenoxy)-5-(2-sulphobenzamido)benzamido]benzamido]-4-[4-(carboxymethoxy)phenylazo]-5-pyrazolone, have the solubilising group separated from the arylazo unit by a connecting chain which can release electrons toward the arylazo unit and so facilitate coupling. C.O.C.

Stable Perhaloaliphatic Carboxylates of Diazo Compounds

National Research Development Corp.

BP 761,054

The salts of diazonium compounds with perhaloaliphatic carboxylic acids are in general only sparingly water-soluble and have good stability in the dry state. Thus *p*-toluidine dissolved in aqueous trifluoroacetic acid is diazotised at $< 10^\circ C$. by addition of sodium nitrite. The precipitated acid salt $CH_3C_6H_4N_2^+ -OOC(CF_3)_3COOH$ may be dried *in vacuo* at $15^\circ C$. to give colourless needles. E.S.

Nickel Salts of Dimethylglyoxime—Red Pigment Stable in Polyvinyl Chloride Compositions

Argus Chemical Laboratory

USP 2,725,365

The nickel salt of dimethylglyoxime is a red pigment which is compatible with polyvinyl chloride resin, neither blooms nor bleeds, and is fast to rubbing. It is made by slowly adding a saturated soln. of dimethylglyoxime to a dilute aqueous solution of a nickel salt made slightly alkaline with ammonia, the pigment being quantitatively precipitated. C.O.C.

Azo Dyes as Ultraviolet Radiation Absorbents in Photographic Emulsions

Kodak

BP 761,728

Dyes of formula $R-N=N-CH(COCH_3)_2$ (R = mono-nuclear aryl) are very good absorbers of ultraviolet radiation, e.g. the dye metanilic acid \rightarrow acetylacetone,

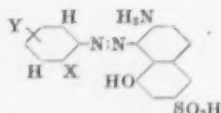
C.O.C.

Red, Water-soluble, Monoazo Dyes for Nylon and Polyacrylic Fibres

ICI

BP 761,468

Monoazo dyes—



(X and Y = CH_3 , Hal , CF_3 , SO_3NH_2 , SO_3Alk , or SO_3NAlk , and are different from one another) give reds on polyamide and polyacrylonitrile fibres, using the cuprous ion method for the latter type of fibre if necessary. The dye obtained by coupling diazotised 4-chloro-2-trifluoromethylaniline under acid conditions with γ acid is particularly valuable for its bluish red shade of good light fastness on nylon.

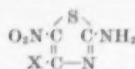
E.S.

Blue Monoazo Disperse Dyes from 2-Amino-5-nitrothiazoles

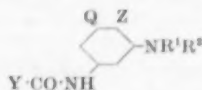
Eastman Kodak Co.

BP 761,792

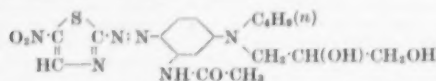
Diazotised 2-amino-5-nitrothiazoles—



(X = H , Alk of 1-6 C, CN , CF_3 , phenyl, p -nitrophenyl or m -nitrophenyl) are coupled with NN' -dialkyl- m -acylaminobenzenes—



(R^1 = hydroxyalkyl of 2-5 C; R^2 = Alk of 1-6 C or hydroxyalkyl of 2-5 C; Y = Alk of 1-3 C; Z = H , OCH_3 , OC_2H_5 , CH_3CO-NH , C_2H_5CO-NH , or C_6H_5CO-NH ; Q = H , CH_3CO-NH , C_2H_5CO-NH , or C_6H_5CO-NH ; one of the members Q and Z must = H) to give disperse dyes, generally of greener shades of blue than the similar dyes of BP 723,933 (J.S.D.C., 71, 261 (1955)) in which the m -acylamino group is not particularly specified. Thus 2-amino-5-nitrothiazole is diazotised by means of nitrosylsulphuric acid mixed with acetic and propionic acids, and coupled with an acid soln. of N - n -butyl- N' - β -hydroxypropyl- m -acylaminoaniline, sodium acetate being added to complete the coupling. The product—



dyes cellulose acetate, nylon, and polyester fibres, deep bright greenish blue which does not redden in artificial light.

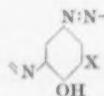
E.S.

Direct Azo Dyes for Aftercoppering

ICI

BP 761,776

The OH group(s) in azo dyes containing at least one group of the formula—



(X = H or Cl , and the N atom *ortho* to the OH group is part of a heterocyclic ring) are sulphated, to give water-soluble direct dyes which may be aftercoppered. The sulphated group is removed during aftercoppering to give an insoluble complex of high washing fastness. Thus the diazo compound benzidine \rightleftharpoons (8-hydroxyquinoline) $_2$ is dissolved in NN -dimethylformamide (I) and sulphated

by adding the addition product of I and SO_3 and stirring at $50^\circ C$. The soluble product dyes cellulose red by the acid aftercoppering method.

E.S.

Metal(Cobalt and Copper)-complex Monoazo Dyes for Anodically Oxidised Aluminium

Ciba

BP 761,348

The cobalt and copper complexes of *oo'*-dihydroxy-monoazo dyes containing at least 2 SO_3H groups, and prepared by coupling a diazotised 2-aminophenol-6-sulphonic acid with a suitable naphtholsulphonic acid or hydroxyquinoline, dye anodised aluminium red to red violet. Thus the monoazo compound 2-aminophenol-4:6-disulphonic acid \rightarrow 2:4-dihydroxyquinoline is treated at $70-75^\circ C$. with aq. copper acetate and copper sulphate for 30 min. The copper complex so formed dyes anodised aluminium bronze, of good light fastness after the usual sealing treatment in boiling water.

E.S.

Red Monoazo Pigments

FBY

BP 761,184

The red pigments obtained by coupling diazotised 2:4-dinitroaniline with *o*-alkoxyarylates of 3:2-hydroxy-naphthoic acid have good fastness to solvents in general and to light. Thus 3:2-hydroxynaphtho-*o*-phenetidine gives a bordeaux, which in cellulose nitrate lacquers is fast to overstripping. The coupling may be carried out in presence of TiO_2 or $BaSO_4$.

E.S.

Red, Chromable, Monoazo Dyes for Wool

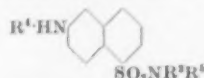
S

BP 761,115

Bright red monoazo dyes which alter only slightly on afterchroming, or may be applied by the metachrome process, are prepared by diazotising amines

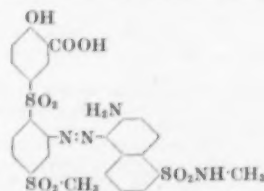


(R^1 = a salicylic acid residue; X = Alk , *cycloalkyl*, *aralkyl*, aryl of benzene series or NR^2R^3 ; R^2 = H or Alk ; R^3 = H , Alk , *cycloalkyl*, *aralkyl*, or aryl of benzene series, R^2 and R^3 together having < 15 C) and coupling with 2-naphthylamine-5-sulphonamides



(R^4 = H , CH_3 , or C_2H_5 ; R^5 = Alk , *cycloalkyl*, *aralkyl* or aryl of benzene series), instead of with 2-naphthylamine-6(or 7)-sulphonamides as described in BP 735,128, J.S.D.C., 71, 680 (1955).

Thus 2-amino-4-methylsulphonyl-3'-carboxy-4'-hydroxydiphenylsulphone is diazotised by running its soln. in aq. NaOH containing sodium nitrite into aq. HCl at < $10^\circ C$. The precipitated diazo compound is coupled with 2-naphthylamine-5-sulphonmethylaniline in presence of HCl to give the monoazo compound—



which dyes wool bright orange red by the metachrome process.

E.S.

Monoazo Disperse Dyes from Cyanoanilines

FBY

BP 760,750

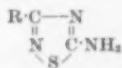
Diazotised cyanoanilines, which may contain Alk or Hal substituents, are coupled with N -hydroxyalkylaniline derivatives to give disperse oranges and reds whose brightness and fastness to light are similar on cellulose acetate and on nylon. Thus the monoazo compound 3-chloro-4-cyanoaniline \rightarrow NN -bis(hydroxyethyl)-*m*-toluidine dyes cellulose acetate and nylon clear yellowish red.

E.S.

Monoazo Disperse Dyes from 5-Aminothiodiazoles
FBv BP 760,61

5-Amino-1:2:4-thiodiazoles—

BP 760,618



(R = H, Alk, OAlk, or Aryl) are diazotised and coupled, preferably with *NN*-dialkylanilines, to give disperse dyes. They are prepared by reacting amidines with thiocyanogen, or by reacting *N*-halogenoacetamidines with inorganic thiocyanates in a solvent, e.g. methanol. Their diazo compounds have great coupling energy and are capable of giving azo compounds with, e.g. *m*-xylene, and anisole. Thus 5-amino-3-methyl-1,2,4-thiadiazol dissolved in sulphuric acid of 40°Bé. is diazotised with aq. sodium nitrite. Coupling with *NN*-diethyl-*m*-chloroaniline gives a bright red, and with anisole a yellow, disperse dye for cellulose acetate. E.S.

Disazo Direct Dyes for Aftercoppering

ICI

BP 760.595

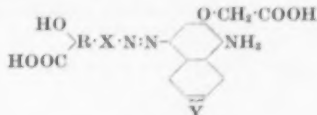
Disazo dyes $R^1 \xrightarrow{\text{alkaline}} J \text{ acid} \rightarrow R^2$ (R^1 = aryl having OH, COOH, or OCH_2COOH *ortho* to its diazotisable NH_2 group; R^2 = an N-containing, metalisable heterocyclic compound) containing $< 3 \text{ SO}_3\text{H}$ groups, yield dyes on cotton whose brightness is generally not much diminished by aftercoppering. Thus 2-amino-6-chlorophenol-4-sulphonic acid is diazotised and coupled with J acid in presence of NaHCO_3 . The aminoazo compound so formed is diazotised and coupled with 4-hydroxyacridine in presence of Na_2CO_3 and β -ethoxyethanol, to give the disazo compound which dyes cotton blue by the aftercoppering process. E.S.

Blue Disazo Direct Dyes for Aftercoppering

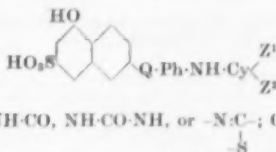
Gy

BP 760,085

Blue direct dyes, whose hue is changed only slightly by aftercoppering, are made by diazotising aminomonoazo compounds—

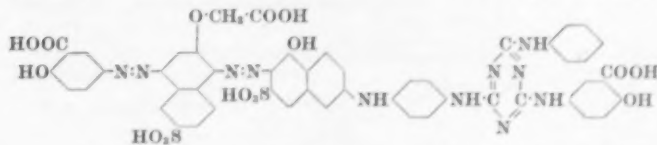


(R = benzene radical in which the OH and COOH groups are *ortho* to each other; X = direct link, $-\text{CO}-\text{Ph}-$, or $-\text{NH}-\text{COPh}-$, where Ph = *m*- or *p*-phenylene radical; Y = H or SO₃H) and coupling with derivatives of J acid



(Q = NH, NH·CO, NH·CO·NH, or $\text{--}\overset{\text{H}}{\underset{\text{H}}{\text{N}}}\text{C--}$; Cy = a 1:3:5-

triazinyl radicle; Z¹ = an organically subst. amine group; Z⁰ = Hal, amino or subst. amino; R and Ph may carry other common substituents). Thus the amino-monoazo compound 5-aminosalicylic acid → 2-carboxy-methoxy-1-naphthylamine-6-sulphonic acid is diazotized and coupled with the condensation product from 1 mol. of cyanuric chloride and 1 mol. each of *N*-*p*-aminophenyl-J acid, 5-aminosalicylic acid, and aniline. The diazo dye so formed—



dyes cotton greenish blue by the aftercoppering process.

E.S.

Orange and Scarlet Disazostilbene Direct Dyes for Aftercoppering

101

RP 760,710

4,4'-Diaminotriblene-2,2'-disulphonic acid (I) or its *N,N*-bis-(*o*- or *p*-aminobenzoyl) derivatives is tetrazotized and coupled with 2 mol. of 1-(5' or 6'-indazolyl)-3-methyl-5-pyrazolone, to give disazo compounds which dye cotton bright orange and scarlet, the hues being little altered by aftercoupping. Thus tetrazotized I coupled in presence of Na_2CO_3 with 2 mol. of 1-(6'-indazolyl)-3-methyl-5-pyrazolone gives a bright scarlet.

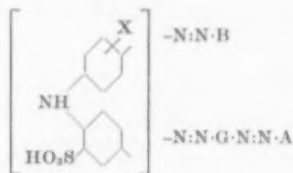
E.S.

Tris- and Poly-azo Dyes for Leather, etc.

CFM

BP 761,800

Tri- and poly-azo dyes—



(X = H, CH₃, OCH₃, Cl, COOH, or SO₃H; A = residue of a diazo component; B = residue of a coupling component; G = residue of a benzene or naphthalene compound capable of coupling in two ways) are obtained by coupling the tetrazo compound of a 4:4'-diaminodiphenylamine-sulphonic acid under acid conditions with 1 mol. of a suitable diamine, dihydroxy, or aminohydroxy compound capable of coupling twice, combining the diazoazo compound so formed with any diazo compound, and finally coupling with any coupling component. The reactions proceed smoothly in spite of the relatively low coupling energy of the tetrazo compounds of 4:4'-diaminodiphenylamine sulphonic acids. The products are red, violet, brown, grey, blue, green, and black dyes for leather. Those having only 2 SO₃H groups in general give good surface dyeing, whereas those with 3 or more SO₃H groups give good penetration of the leather. Some of the products dye cellulose, and may be metallised if appropriate groupings, e.g. a salicylic acid residue, are present.

Thus 4:4'-aminodiphenylamine-2-sulphonic acid is tetrazotized and acid-coupled with 1 mol. of 1-amino-8-naphthol-4-sulphonic acid. The diazonzo compound is filtered off, and coupled with diazotised sulphonic acid, soda ash being added, and when this coupling is complete, an aq. soln. of 1 mol. of resorcinol is added. The triazo compound so formed dyes leather a well-penetrated black.

E.S.

Navy Blue, Grey, and Black Copperable Polyazo Direct Dyes

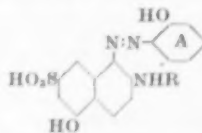
5

BP 760,705

Tetrazotised benzidine derivatives—



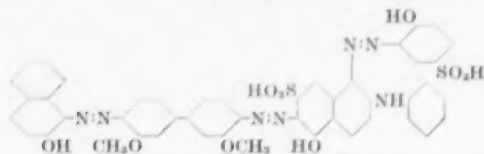
(X = OH, OCH₃, OC₂H₅, OCH₂COOH, or COOH) are coupled with 2 mol. of compounds—



(R = H, Alk, hydroxyalkyl, cycloalkyl, aralkyl, or aryl;

A may contain substituents other than a carbonyl group) or with 1 mol. of such compound and 1 mol. of a suitable coupling component which couples in the *ortho* position to an OH group and/or contains an *o*-hydroxycarboxy grouping, to give direct dyes which may be coppered on the fibre or in the dyebath. Thus tetrazotised dianisidine and soda ash with 1 mol. of the acid obtained by the acid coupling of

diazotised 2-aminophenol-4-sulphonic acid with *N*-phenyl-*J* acid, and a soln. in aq. NaOH of β -naphthol is then added to give the triazo dye—



which dyes cellulose navy blue by the single bath or 2-bath coppering process. E.S.

Orange and Scarlet Azoic Dyes for Cellulose Acetate and Nylon

FH BP 761,067
Orange and scarlet azoic dyeings fast to cross-dyeing are produced on cellulose acetate and nylon by combining on the fibre the diazo compound from an aminophthalic dialkyl ester with an aryldide of 3:2-hydroxynaphthoic acid having an OAlk group in the aryldide residu. Thus 3:2-hydroxynaphtho-*o*-anhydride and 4-aminophthalic acid diethyl ester are applied together to acetate rayon at 75°c. Diazotisation is carried out in a cold nitrous acid bath, and development in a slightly alkaline bath at 60–80°c., to give the orange dye. E.S.

Claret and Red Azoic Dyes

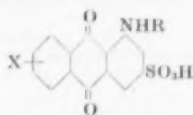
FH BP 760,574
N-Acetoacetyl-*p*-xylylidine gives bright red azoic dyeings and prints with diazotised 4-amino-2:5-dimethoxy-3'-nitroazobenzene, and clarets with diazotised 4-amino-2:5-dimethoxy-4'-nitroazobenzene. Pale dyeings of these combinations maintain the same hue as the deeper dyeings, whereas *NN*-bisacetoacetyl-*o*-tolidine gives with these diazo compounds reds in full depth but oranges in pale dyeings and prints. E.S.

Khaki Azoic Dyes for Camouflage

FH BP 760,371
The azoic dyes or prints obtained by coupling the *q*-naphthylamide of 3-hydroxydiphenylene oxide-2-carboxylic acid on the fibre with diazotised 2-amino-4-nitrotoluene or with a diazotised 2:5-dihalogenoaniline are respectively brownish khaki and greenish khaki, giving in infrared photography an effect similar to that of chlorophyll and hence suitable for camouflage purposes. E.S.

Anthraquinone Acid Dyes

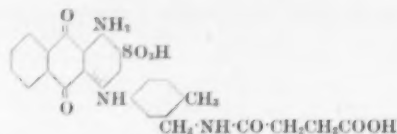
FH BP 761,382
Bluish-red neutral dyeing acid dyes of formula—



(R = completely or partially hydrogenated phenyl or naphthyl; X = H or SO₃H) are made either (a) by condensing a 1-bromoanthraquinone-3-sulphonic acid with an appropriate amine, or (b) by condensing a 1:3-dibromoanthraquinone with an appropriate amine and replacing the 3-Br with SO₃H by treating with e.g. Na₂SO₃ or (c) by condensing 1-amino-4-bromoanthraquinone-2-sulphonic acid with an appropriate amine and removing the 1-NH₂ by diazotising. R.K.F.

Blue Anthraquinone Acid Dyes

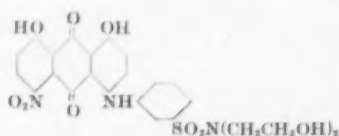
General Aniline USP 2,722,534
Treating 1-amino-4-bromoanthraquinone-2-sulphonate with an aromatic amine having an acylated aminomethyl group yields a blue acid dye. Thus *N*-(5-amino-2-methylbenzyl)succinimide is heated in 10% aq. NaOH until dissolved. The solution is cooled and enough dil. HCl added to cause complete precipitation, the precipitate filtered off and washed neutral. The filter cake was stirred for 16 hr. at 80°c. with Na-1-amino-4-bromoanthraquinone-2-sulphonate, Na₂CO₃, CuCl₂ and water. 20% Brine was then slowly added and the mixture allowed to cool to room temperature when a blue acid dye settles out. It has the formula—



and yields greenish-blue dyeings of excellent fastness to light. C.O.C.

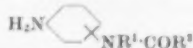
Nitroanthraquinone Disperse Dyes of Excellent Fastness to Gas Fume Fading

General Aniline USP 2,722,535
Introduction of a substituted sulphonamide group in either *o*-, *m*- or *p*-position of the benzene nucleus of the anilino group attached to the 4-position of either 4:5-dinitrochrysazin or 5:8-dinitroanthraquinone yields blue disperse dyes for acetate rayon and Terylene, the dyeings having excellent fastness to gas fume fading. Thus *N*'-bis(2-hydroxyethyl)metanilamide, 4:5-dinitrochrysazin stirred together for 9 hr. at 140°c. in nitrobenzene, cooled, water added and the solvent steam distilled off, yielded the dye—

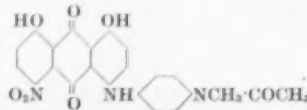


which dyes acetate rayon a reddish blue of good fastness to washing, light and burnt gas fumes.

USP 2,722,536
Condensing 4:5-dinitrochrysazine or 4:8-dinitroanthraquinone with compounds of formula—



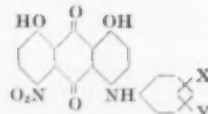
(R¹ = Alk; R² = H or Alk) yields dyes having excellent affinity for cellulose acetate. Thus condensing 4:5-dinitrochrysazine with *p*-amino-*N*-methylacetanilide yields the dye—



It gives heavy blue dyeings on cellulose acetate which have excellent fastness to light and gas fume fading. C.O.C.

1:8-Dihydroxy-5-nitro-4-anilinoanthraquinone Compounds—Blue Disperse Dyes

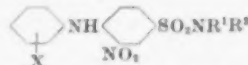
Eastman Kodak Co. USP 2,726,251
Dyes of formula—



(X = mono- or di-hydroxyalkyl of 3 C, (OCH₂CH₂)_n-OH (n = 1–4) or OCH₂(CH₂)_m-O-R (m = 1–3); Y = H, Cl, OH, CH₃O or CH₃) give blue dyeings on cellulose esters of outstanding fastness to light and gas fume fading. C.O.C.

2-Nitro-4-sulphonamidodiphenylamine Dyes for Acetate Rayon

Eastman Kodak Co. USP 2,725,390
Compounds of formula—



(X = phenyl; R¹ and R² = H, Alk of 1–4 C, hydroxyalkyl of 1–4 C; alkoxyalkyl of 3–6 C or monohydroxyalkoxy of

3-6 C), e.g. 2-nitro-4-sulphamido-4'-phenyldiphenylamine, yield yellow dyes of excellent fastness to light and gas fume fading on acetate rayon and similar materials.

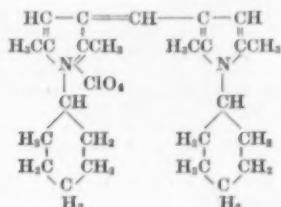
C.O.C.

Pyrrole Dyes

Eastman Kodak Co.

USP 2,725,378

Condensation of a pyrrole base with formic acid yields a monomethine cyanine dye containing a pyrrole nucleus. Thus 1-cyclohexyl-2,5-dimethylpyrrole condensed with formic acid in presence of perchloric acid yields the orange dye—



It is soluble in ethanol or CHCl_3 , is completely decolorised by weakly alkaline solutions and is photographically inert.

C.O.C.

Anthraquinone Diacridone Vat Dyes

DuP

USP 2,726,242

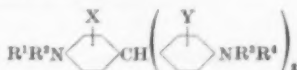
Anthraquinone-1:5-dianilido- α : α' -dicarboxylic acid heated at 150–170°C. with benzoyl chloride and phosphoric acid gives a good yield of the corresponding anthraquinone diacridone of high quality as a vat dye. C.O.C.

Leuco Triarylmethane Compounds

DuP

USP 2,726,252

Compounds of formula—



(R^1 , R^2 and R^3 = Alk, β -hydroxyethyl or β -organoethyl; R^4 = Alk, benzyl, β -hydroxyethyl or β -cyanoethyl; X and Y = H, Hal or CH_3) are obtained in high purity and free from the corresponding carbinol and dye (salt form) by reacting 1 mole of a *p*-aminobenzaldehyde whose amino H atoms are replaced by Alk, β -hydroxyethyl or β -cyanoethyl, with 2 mol. of a *tert*. arylamine at 70–120°C. in presence of urea, thiourea or a derivative thereof in which hydrogen, on one of the N atoms only, is replaced by inert substituents and a strong water-soluble acid. Incorporated into paraffin wax and stabilised with chlorine they are used for carbon paper coatings or in colourless hectograph inks.

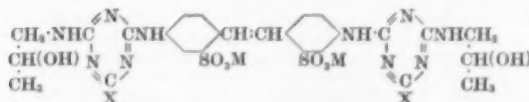
C.O.C.

Fluorescent Brightening Agents of the Stilbene Series

S

BP 760,082

Reacting 2 mol. cyanuric chloride or cyanuric bromide in any order with 1 mol. 4:4'-diaminostilbene-2:2'-disulphonic acid, 2 mol. 1-amino-2-propanol and 2 mol. ammonia and/or a primary amine yields compounds of formula—



(M = H or cation; X = same or different NH_2 or aryl-amino). They are fluorescent brightening agents of good affinity for vegetable, animal and polyamide fibres. They impart bluish fluorescence.

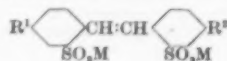
C.O.C.

Water-soluble Derivatives of 4:4'-Diaminostilbene—Fluorescent Brightening Agents

FBY

BP 760,008

Compounds of formula—



(R^1 = R^2 , 2:4-diamino-1:3:5-triazyl-(6-amino, 1:2:3:4-tetrahydro-6-(4'-Na sulphonate-*n*-butoxy)-1)-7-naphthoyl-amino, a phenylcarbamido group or α -pyrrolidone-*N*-(γ -ureido-*n*-propylene)), e.g. di-Na salt of 3:4'-di-(1:2:3:4-tetrahydro-6-methoxy-7-naphthoylamino)-stilbene-2:2'-disulphonic acid, are fluorescent brightening agents especially useful on cellulose. They may be used in detergents.

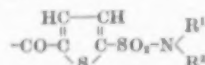
C.O.C.

Acylamino Vat Dyes containing a Sulphamylthienoyl Group

Ciba

BP 761,066

A 1-amino-4-acylamino-6:7-dihalogenoanthraquinone is acylated using starting materials which give a product containing the acyl residue



(R^1 and R^2 = Alk or the C's of a ring). The red to violet compounds so obtained are vat dyes. Thus 2-carboxy-thiophene-5-N:N-dimethylsulphonamide is heated to 90°C. in nitrobenzene with SOCl_2 and 1-amino-4-benzoyl-amino-6:7-dichloroanthraquinone added, heating continuing at 120°C. for 2 hr.

R.K.F.

Dispersion of Phthalocyanine Pigments

Gy

BP 761,308

A phthalocyanine is ground in a ball mill with a soluble salt, e.g. anhyd. sodium acetate, and a solvent of m.p. > 20°C., e.g. naphthalene, the solvent removed, e.g. by steam distilling, and the salt by filtering and washing to produce the stable β -modification in finely dispersed form.

R.K.F.

Phthalocyanine Sulphur Dyes

CFM

BP 761,287

A phthalocyanine is heated at 100–150°C. with hydroxylamine and ClSO_3H in the presence of a compound of a group Va or VIA element, e.g. ammonium molybdate, to give grey via to light sulphur dyes.

R.K.F.

Triangulene Compounds

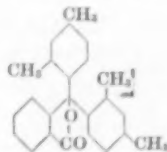
Petrochemicals Ltd.

BP 760,912

Carboxylic acids of the 4:8-quinone of triangulene—



which on sulphonating give blue chrome dyes, are made by a series of reactions from bis-(*m*-xylyl)- and bis-(*p*-xylyl)-phthalides. Thus bis-(*m*-xylyl)phthalide



is first reduced to the corresponding 2-carboxy-tetramethyltriphenyl methane with Zn and alkali. This is then converted to the dimethyl-xylyl-anthrone in a $\text{ZnCl}_2/\text{NaCl}$ melt. Oxidation follows, first with dil. HNO_3 and then with alkaline KMnO_4 . The resulting 9-phenyl-oxanthranol tetracarboxylic acid is reduced to the corresponding 9:10-dihydroanthracene, which is finally cyclised in H_2SO_4 to triangulene-4:8-quinone-6:10-dicarboxylic acid. The 7:11-dicarboxylic acid is obtained by similar methods from bis-(*p*-xylyl)-phthalide.

R.K.F.

Wood Stains containing a Flavonone Azo Dye and a Fluorescent Brightening Agent

Chadeloid Corp.

USP 2,723,898-9

Coupling citrus flavanone derivatives, e.g. hesperetin or naringin found in citrus pulp and citrus waste, with diazotised aniline or aniline bases yields water soluble dyes having excellent affinity for wood. Application of the hot aqueous solution to wood gives good coloration without

affecting the grain. Their brightness may be improved by adding a fluorescent brightening agent to the solution.

C.O.C.

Esters of Bixins and norBixins—Fat and Food Colours

Roche Products

BP 760,346

Esters of bixins and norbixins with alcohols of > 4 C are very soluble in fats, oils and fat solvents. Thus amyl bixin is 10 times and octylbixin 20 times more soluble in fat solvents than in butyl bixin. The vitamin A esters of bixins and norbixins are especially valuable as, apart from their excellent solubility in fats, they impart a very stable colour to foodstuffs in which they are incorporated. 17 Examples of the preparation of such esters are given.

C.O.C.

Red Iron Oxide

C. K. Williams & Co.

BP 762,102

Colloidal seeds of hydrous Fe_2O_3 are formed by oxidising the precipitate formed from mixing solutions of ferrous salt and alkali and then the growth of red Fe_2O_3 is effected in the seed. Growth is continued until the particles have the desired colour. There must be 0.9–1.1 equivalents of alkali to each equivalent of iron salt, as outside these limits a yellow oxide of Goethite structure is formed. The product is a rich red pigment which can be used without the aid of an organic toner.

C.O.C.

Titanium Dioxide

British Titan Products Co.

BP 761,770

TiCl_4 is air oxidised by feeding it to a fluidised bed of inert material of particle size 40–1000 μ at 700–1200°C.

C.O.C.

Dry Pigments of Improved Dispersibility

Allied Chemical & Dye Corp.

BP 760,116

Activated silica gel of surface area < 100 sq.m./g., oil absorption value < 100 and average particle size 0.01–50.0 μ , is mixed with a finely divided organic dye or pigment dispersed in liquid and then the liquid removed and the pigment ground. The products are readily dispersed in varnish and yield a paint of greater tinctorial strength than does the same amount of organic colouring material obtained by grinding filter cake dried in absence of activated silica gel.

C.O.C.

Azoic Red Pigments (V this page)

Pigment Properties and Printing Ink Behaviour (V this page)

p-Diamines in Hair Dyes (VIII p. 72)

Disperse Dye Mixtures (VIII p. 73)

Blue and Violet Tetra-azaporphin Dyes for Polyesters (VIII p. 73)

Copolymers of Allyl Alcohol and Acrylonitrile produced by Dye-sensitised Photopolymerisation (XIII p. 78)

Coloured Polyphosphate-Dye Polymers (XIII p. 78)

V—PAINTS; ENAMELS; INKS

Electrical Charges of Pigments in Binder Systems

G. Florus and K. Hamann

Farbe u. Lack, 62, 260–7, 223–231 (1956);

Chem. Abs., 50, 16129 (10 Nov. 1956)

Inorganic pigments act essentially alike in the same binder, being positive, negative or electrically neutral according to the surface-activity. The charge on organic pigments is independent of the vehicle. By adding wetting and flow agents and proper selection and combination of binders the electric charge on inorganic, but not on organic, pigments can be controlled. There is close relationship between the charge on the dispersed pigments and the stability of the dispersion. Hence important properties such as degree of dispersion, gloss, hiding power, instability, flow, and pigment-absorption power, are connected with the electric charge.

C.O.C.

Azoic Red Pigments

D. H. Stubbs

Am. Ink Maker, 34, (7), 30–2, 61 (1956);

Chem. Abs., 50, 16129 (10 Nov. 1956)

The properties of these pigments are described from the ink makers' standpoint. They are recommended for printing soap wrappers because they have good alkali resistance and low solubility in water and soap. C.O.C.

Pigment Properties and Printing Ink Behaviour

N. Holtzman

Am. Ink Maker, 34 (4), 32–7, 67, 69 (1956);

Chem. Abs., 50, 16130 (10 Nov. 1956)

Fundamental pigment properties are discussed with the aid of electron micrographs and charts showing various printing ink properties as a function of pigment particle size for Lithol Reds and Rubines and for rosinated and nonrosinated Benzidine Yellows.

C.O.C.

Pigment Dispersion in Acrylic Paint Systems

G. Allyn and G. J. Scheiffele

Offic. Dig. Federation Paint & Varnish

Production Clubs, 27, 520–8 (1955);

Chem. Abs., 50, 16129 (10 Nov. 1956)

The fluidity titration method is suitable for determining the activity of dispersing agents in preparing pigment dispersions for water-based paint systems. Tube colours, although usually available as oil dispersions, can be adapted for use in tinting white water-based systems.

C.O.C.

Aluminium Foil and Requirements of Inks for Offset Foil Printing

D. Pingree

Am. Ink Maker, 34 (8), 30–2, 67 (1956)

Effect of the Type and Amount of Pigment on the Permeability of Coating Films

H. Berger

Deut. Farben-Z., 10, 211–5, 243–251 (1956)

PATENTS

Preventing Discoloration of White or Light-coloured Paints on Rubber

Dunlop Rubber Co.

BP 762,573

The yellowing or browning undergone on storage by white or light-coloured paints applied to rubber is prevented by use of an undercoating or pigmented outer coating containing cyclised rubber.

C.O.C.

Tinting Compositions for Paints

Lewis Berger & Sons

BP 762,439

Tinting compositions suitable for colouring both oleo-resinous enamels and water-thinned emulsion paints comprise a water-insoluble pigment, a volatile organic solvent—at least some of which is soluble in oleo-resinous varnishes and insoluble in water, and a liquid surface-active agent, some of which at least is nonionic.

C.O.C.

Corrosion-resistant Wrinkle Coating Compositions containing Titanium or Titanium Alloy Powder

New Wrinkle

USP 2,725,363

Aqueous Dispersions of Inorganic Pigments

Rohm & Haas Co.

BP 758,986

An inorganic pigment is dispersed in water by aid of a water-soluble salt from a copolymer of maleic anhydride and an olefinically unsaturated hydrocarbon of 5–10 C containing only one active olefinically unsaturated group, the copolymer preferably having mol. wt. 750–5000. This enables fluid dispersions of high solids content to be obtained. They are used to colour aqueous resin compositions.

C.O.C.

Powdered Colour Base for Paints, etc.

Seidlitz Paint & Varnish Co.

USP 2,722,485

A resin, pigment and other components of the colour base are mixed with a water-soluble solvent and then led into water to produce a precipitate, which is dried and sifted. The product is added to the paint to colour it.

C.O.C.

Self-hardening Lacquers or Paints

M. J. Ringstrom

BP 760,138

Derivatives of benzene, e.g. benzaldehyde, which are inactive as hardeners in absence of air, but which in contact with air react at room temperature to yield an acid hardening product are used in urea-formaldehyde resin paints as hardening agents. Paints containing them can be stored indefinitely in absence of air.

C.O.C.

Diatomaceous Calcite as an Extender Pigment for Paint (IV p. 65)

VI—FIBRES; YARNS; FABRICS

Table of Fibre Properties

A. Wilhelm

Textil Praxis, 11, (facing p.) 1036 (Oct. 1956)

Viscoelastic Properties of Regenerated Cellulose Filaments spun under Various Degrees of Stretching

K. Fujino, H. Kawai, T. Horino, and K. Miyamoto

J. Soc. Textile Cellulose Ind. Japan, 12, 639-646 (Sept. 1956)

The viscoelastic behaviour of regenerated cellulose filaments spun under various degrees of stretching was measured under normal atmospheric conditions and under conditions of swelling by water by using several longitudinal vibration methods over a range of frequencies from subsonic to supersonic. The results were represented in terms of relaxation spectrum over a range of relaxation times from 1×10^{-6} to 1 sec. The relaxation spectrum in the time range longer than that covered here may be very much decreased by swelling in the order of decreasing orientation factor, while the spectrum in the time range covered here is not so much affected. Such a remarkable decrease of relaxation spectrum in the longer time range must be ascribed to destruction of hydrogen bonds and to increase of chain mobility in the amorphous region due to swelling by water.

AUTHORS

Cellulose Triacetate

A. Sippel

Textil Praxis, 11, 1016-1019 (Oct. 1956)

A review of the manufacture, heat setting, dyeing, and identification of triacetate fibres.

B.K.

Yellowing of Wool by Thermal Degradation

G. Mazingue and M. van Overbèke

Bull. Int. Text. France (62), 27-36 (Aug. 1956)

Wool, Ardil, and Merinova, which contain a high proportion of basic amino acids, show a much greater tendency to yellow on heat treatment than do silk and Vicora, which are relatively poor in basic amino acids. Treatment of wool at 160°C. for 7 hr. reduced the content of side-chain amino groups by approx. 30%, as determined by the fluorodinitrobenzene (FDNB) method. J.C.F.

Reactivity of Amide Groups in Acid Hydrolysis of Polyamides from ϵ -Aminocaproic Acid

D. Heikens

J. Polymer Sci., 22, 65-70 (Oct. 1956)

The rate constant for the hydrolysis of amide groups in dimers, trimers, and oligomers is found to be independent of chain length, showing that the amide groups are equally reactive.

W.R.M.

Effect of Nuclear Radiation on Fibrous Materials. I—Dacron Polyester Fibre

O. Tenzler and H. A. Rutherford

Text. Research J., 26, 796-801 (Oct. 1956)

Dacron filaments exposed to thermal neutron and γ irradiation showed a slight degree of cross-linking, evident in an increased modulus of elasticity. The effect was dependent on the degree of orientation of the fibre, the optimum draw ratio lying between 2.5:1 and 3.0:1. At higher draw ratios, degradation reactions, probably chain scission, predominate.

J.C.F.

Teflon Tetrafluoroethylene Fibre

J. T. Rivers and R. L. Franklin

Text. Research J., 26, 805-811 (Oct. 1956)

A survey of preliminary use-development investigations of Teflon fibre. The chief uses envisaged are—packing materials for pumps and valves, filter fabrics, gasketing materials, and laundry fabrics. In such applications the unique corrosion resistance and heat stability of Teflon may lead to sufficiently improved process operation and maintenance reduction to offset its comparatively high cost.

J.C.F.

PATENTS

Cellulose Fibres containing Ether Groups and β -Propiolactone Substituents

U.S. Secretary of Agriculture

USP 2,724,633

Cellulose fibres are etherified with either aminoalkyl sulphuric acid, monochloroacetic acid, alkylene oxide, acrylonitrile or alkyl sulphate (Alk in each case of 1-3 C) until 0.1-2.5 of etherifying radical is taken up per anhydroglucose unit. The etherified fibres are then treated with β -propiolactone until they contain 0.5-26.0% of their weight of β -propiolactone substituents. The treated

fibres have wool-like properties, and are resistant to degradation by heat or acid and to creasing. C.O.C.

Blends of "Sativa" Treated Bast Fibres and Natural or Synthetic Fibres

A. F. Burkardt

BP 761,559

Yarns having a predetermined relative wet strength differing from the normal dry or wet strength of the component fibres are produced by determining the relative wet and dry strength of at least two fibres, natural or synthetic, one of these fibres being "Sativa" treated natural fibre or yarn which comprises either (a) Sativa, i.e. natural bast fibres such as hemp, flax or ramie, degummed and bleached and thereby preshrunk or (b) nylon, wool or cotton with Sativa or (c) bright rayon tow or "Aralac" with Sativa, and spinning such fibres together to produce a composite yarn. A mathematical formula is given relating the strength of the composite yarns to their composition. Fabrics produced from these yarns maintain their normal dry strength when laundered, and can be made stronger wet than dry.

W.G.C.

Rendering Cyanoethylated Cellulose Soluble in Dilute Alkali

Londat Aetz Fabric Co.

USP 2,724,632

When cyanoethylated cellulose is treated with chromic or periodic acid the product is soluble in alkali of all strengths. The insoluble residue is quite small and is not gummy or sticky. Cyanoethylated cotton so treated is very suitable as a backing fabric in the manufacture of lace, as it is readily removed by treatment with dilute alkali.

C.O.C.

Supertwisting and Dyeing Polyamide and Polyester Filament Yarns

Belmont Throwing Co.

USP 2,725,714

A multifilament yarn is twisted in one direction, after which it is preset, dyed, twisted further in the same direction, set and preshrunk. Then, if desired, additional twist is given to the yarn in which case the yarn must be again set and dried. The yarn is then untwisted either to zero point or even given a reverse twist and is then plied or twisted with a yarn treated in a similar manner but twisted in the opposite direction.

C.O.C.

Stabilised Polyester Fibre

ICI

BP 758,684

Treating polyester fibre with a rubber antioxidant preserves its tensile strength when it is heated in air at a high temperature, the process being particularly applicable to the fibres produced by the method described in BP 682,866.

C.O.C.

Acrylonitrile Copolymer Solutions

Vereinigte Glanzstoff-Fabriken A.G.

BP 759,342

Solutions of acrylic polymers suitable for spinning are prepared by adding the polymer to a solvent boiling at 100°C. or higher under vacuum to de-aerate the solution.

W.G.C.

Spinning Solutions of an Acrylic Copolymer

American Cyanamid

BP 759,095

A clear homogeneous solution suitable for spinning comprises (a) a ternary polymer containing methyl acrylate, vinyl acetate, acrylamide and/or methacrylamide (2-15%), vinyl pyridine (1.5-15%) and the remainder acrylonitrile and (b) a solvent which is an aqueous solution of water-soluble salt having highly hydrated ions in solution.

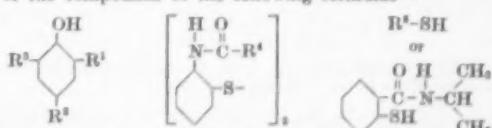
W.G.C.

Colour Stabilisation of Acrylonitrile Polymers

American Cyanamid

BP 761,685

Discoloration of acrylonitrile copolymers by heat is prevented by adding 0.001-0.3% by weight of one or more of the compounds of the following formulae—



(R¹, R² and R³ = Alk, at least one containing 3 C and is ortho to the OH group), e.g. 2-isopropyl-4:6-dimethyl phenol; (R⁴ = Alk of 2-12 C or Ar of 6-12 C), e.g. α -dibenzamido-diphenyl disulphide; (R⁵ = Ar of 6-12 C), e.g. thiophenol, and the organic amine or bivalent metal salts thereof.

W.G.C.

Acrylic Fibres of Improved Dyeing Affinity

Dow BP 758,937
At least 1% of a water-soluble dyeing assistant is applied to the acrylic fibre freshly spun from an aqueous saline solution into an aqueous coagulant, and fixed by drying the treated fibre.
W.G.C.

Acrylonitrile-Itaconic Ester Polymers

Chemstrand BP 762,390
Polymers made from acrylonitrile, aminoesters of itaconic acid and, if desired, one or more other ethylenic compounds have good affinity for basic and disperse dyes.
C.O.C.

Improving the Abrasion Resistance of Fibres and Films

Deutsche Gold- und Silber-Scheideanstalt BP 759,374
The abrasion resistance of synthetic fibres and films is improved by incorporating in the spinning dope 0.1–2% of an aerogel of a metal oxide, e.g. silica and titanium dioxide.
W.G.C.

Pile-surfaced Materials

BrC BP 762,467
A flock of loose fibres is fed between an adhesive-coated surface and another surface, one of the surfaces being that of a sheet of insulating material. This sheet is frictionally electrified to produce an electric field across the space between the two surfaces so as to orient the fibres and project them onto the adhesive. Finally the adhesive is hardened.
C.O.C.

Properties and Dyeing Behaviour of Cellulose Triacetate (VIII this page)

Blue and Violet Tetra-azaporphin Dyes for Polyesters (VIII p. 73)

Increasing the Dye-receptivity and Moisture Absorption of Acrylonitrile Polymers (VIII p. 73)

Effect of Yarn Twist on Fabric Crease Recovery (X p. 75)

Phase State of Cellulose in Oriented Fibres (XI p. 76)

Polyacrylonitrile: Molecular Weight-Viscosity Relation (XIII p. 78)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING**Recent Work on Surface Activity, Wetting, and Dewetting**

J. Guastalla

J. Colloid Sci., 11, 623–636 (Oct. 1956)

A review of recent work.

W.R.M.

Foaming of Aqueous Soap Solutions—Chemical Composition of the Adsorption Layer of Aqueous Solutions of Sodium Oleate

M. Raison

World Congress on Surface-active Agents, 1, 105–113 (1954)

Thermodynamics of the Surface Interactions of Ionic Detergents

B. E. Pethica

World Congress on Surface-active Agents, 1, 79–83 (1954)

Influence of Added Electrolytes on Aqueous Dodecylammonium Chloride Solutions

D. N. Eggenberger, C. W. Hoerr, A. E. Brake, A. Veis, and H. J. Harwood

World Congress on Surface-active Agents, 1, 72–78 (1954)

Foaming Power of Soaps of Fatty Acids as a Function of pH and Concentration

D. G. Dervichian

World Congress on Surface-active Agents, 1, 125–129 (1954)

Liquid Detergents in Automatic Laundering Processes

F. G. Edis-Bates and R. C. Tarring

World Congress on Surface-active Agents, 2, 646–648 (1954)

Recent Advances in the Use of Sodium Carboxymethylcellulose as a Detergent Aid

F. J. Pollok

World Congress on Surface-active Agents, 2, 689–691 (1954)

A summary is given of recent work on the effect of the degree of substitution of sodium carboxymethylcellulose (I) on soil-suspending power, the use of I with soap and with synthetic detergents, in special detergent compositions, and as a soil-resistant finish. A method is described for the estimation of I in detergent compositions.

W.K.R.

Some Aspects of the Adsorption of Anionic Surface-active Agents by Cotton

S. R. Epton and J. M. Preston

World Congress on Surface-active Agents, 2, 471–477 (1954)

Adsorption of sodium *sec.*-alkyl sulphates (I) (in the absence of electrolytes) is shown to be due to the non-cellulosic component, and is proportional to the N content of the cotton. Purified cotton adsorbs I in the presence of electrolytes, and theoretical treatment of the experimental data shows that the adsorption is physical and non-specific.
W.K.R.

Detergents in Sheepskin Scouring

F. G. Edis-Bates and R. C. Tarring

World Congress on Surface-active Agents, 2, 562–563 (1954)

The treatments to which sheepskins are submitted from the raw skin to the dyed and finished product are described briefly, and the advantages in using synthetic detergents in various processes are indicated.
W.K.R.

Role of Detergents in Raw-wool Washing

E. G. Carter

World Congress on Surface-active Agents, 2, 442–450 (1954)

Special problems arising in raw-wool scouring are—the large amounts of grease and soil to be removed, the vulnerability of the fibres to alkali and mechanical damage, and the necessity of leaving the wool with the correct pH and grease and detergent contents to suit subsequent processing. The relative merits of soap and synthetic detergents in meeting these requirements are discussed.
W.K.R.

PATENTS**Diminishing Formation of Chlorine Dioxide in Acid Chlorite Baths**

FB BP 760,206

NH_4Cl , $(\text{NH}_4)_2\text{SO}_4$, NH_4ClO_3 or an NH_4 salt of an aliphatic carboxylic acid of 1–2 C and a sulphonic and/or carboxylic acid containing an aliphatic radical of > 7 C, is added to the bath. Thus when bleaching viscose staple fibre at 70°C. in 0.1% aq. Na chlorite using a goods to liquor ratio of 1 : 20, addition of 0.35 g./l. oleyl sarcoside and 0.6 g./l. of NH_4Cl yielded a better white and used 30% less chlorite.
C.O.C.

Bleaching of Fur Felt Hoods

ICI

BP 760,509

The time needed for bleaching can be considerably shortened by using neutral or faintly acid solutions of hydrogen peroxide or per-salts at 70–100°C.
C.O.C.

VIII—DYEING**Properties and Dyeing Behaviour of Cellulose Triacetate**

K. Dellian

Textil Praxis, 11, 1011–1016 (Oct. 1956)

The properties of cellulose triacetate are described with special reference to heat resistance and effects of organic solvents, acids, alkalis, and oxidising agents. The dyeing of triacetate and unions thereof with other fibres are detailed referring to acid, direct, vat ester, leuco vat, basic, disperse, and developed dyes and also to pigment-padding processes.
B.K.

p-Diamines in Hair Dyes

R. Heilingötter

Fette, Seifen, Anstrichmittel, 56, 610–4 (1954);

Chem. Abstr., 50, 16046 (10 Nov. 1956)

The toxicity of hair dyes containing p-phenylenediamine is greater than that of those containing p-toluylenediamine. As the latter have less colour value than the former it is necessary to use 50% more to get the same depth.
C.O.C.

Dyeing of 6-Nylon with Disperse Dyes

M. Yoshida and M. Nishio

I—Kinetics of the Dyeing Process

J. Soc. Textile Cellulose Ind. Japan, 12, 652–657

(Sept. 1956)

Rate-of-dyeing isotherms at 40°, 60°, 80°, and 100°C., covering the first hour of dyeing, have been determined for four different commercial disperse dyes on 6-nylon fabric. The curves can be expressed approximately as hyperbolas and a velocity coefficient was derived from the best fitting hyperbola. The values of the equilibrium exhaustion and the velocity coefficient of dyeing may be used to select

dyes which are compatible in mixtures. The effect of temperature on dyeing rate is discussed. The rate of dyeing varies exponentially with the reciprocal of the absolute temperature in the same way as most chemical reactions or diffusion processes, and the activation energies of the dyeing process for four dyes lie in the region of 22–26 kcal./mole. By a graphical method it is possible to predict the course of dyeing with varying temperature, and this has been confirmed experimentally.

II—Dyeing Properties of Commercial Disperse Dyes

Ibid., 657–661

The technical velocity coefficient, time of half-dyeing, and percentage exhaustions at equilibrium were obtained for 22 commercial disperse dyes from their rate-of-dyeing curves at 80°C. From the foregoing it is evident that they will provide much useful information on the behaviour of the disperse dyes on 6-nylon which will be of practical value in assessing comparative rates of dyeing and selecting compatible dye mixtures.

AUTHORS

Dyeing of Nylon with Acid Dyes. I—Method of Measurement of Dye Diffusion within the Unstretched Nylon Gut

M. Yoshida and H. Hirota

J. Soc. Textile Cellulose Ind. Japan, 12, 662–664 (Sept. 1956)

The diffusion rates of several acid dyes were determined using the unstretched nylon gut. The method consists in measuring the dye penetration from microscopical observation of the ring-dyed gut section. The distance of penetration is proportional to the square root of dyeing time, and in general increases as the dye concentration increases and the pH of the dyebath decreases. From the temperature dependence of diffusion rate the apparent activation energy of diffusion is calculated. The diffusion rate varies from dye to dye, but its temperature dependence remains almost the same.

AUTHORS

Resistance of Dyes used on Synthetic Fibres to Heat Setting and the Thermosol Process

F. Weber

Textil Praxis, 11, 1034–1036 (Oct. 1956)

Fastness figures are given for ranges of dyes manufactured by Gy, DuP, BASF, and FBy, used on polyamide, polyester, and polyacrylonitrile fibres, when subjected to the Thermosol and heat-setting processes. These results relate to both colour change and sublimation on to white materials.

B.K.

PATENTS

Dyeing or Printing Cellulose with Disazo or Polyazo Dyes containing Copper bound to o-Hydroxyazo Groups

Ciba

BP 763,071

Sparingly soluble copper compounds of dyes containing more than one azo group, the copper being bound exclusively to o-hydroxyazo groups, at least one of which has no sulphonic acid group *ortho* to the OH group, are readily dyed or printed onto cellulose from neutral or alkaline baths or pastes containing an amine, advantageously an aliphatic amine, having at least one amine group separated from OH by 2 C atoms, and a compound containing anions of a phosphoric acid. The copper compound should be such that after being separated from an alkaline medium at least 90% of it should dissolve in 50 times its weight of boiling water and twice its weight of 1:2-di-(β -oxy-ethylamido)ethane.

C.O.C.

Disperse Dye Mixtures

Ciba

BP 762,538

Mixtures of 1:4-diaminoanthraquinone and 1:4-di-(alkylamino)anthraquinone produce dyeings which in strength and purity closely approach those made with the corresponding 1-amino-4-alkylaminoanthraquinone. Addition of a small amount of a 1-amino-4-alkylaminoanthraquinone to the above mixture produces a result equal to or better than that produced by use of 1-amino-4-alkylaminoanthraquinone alone. Thus a mixture of 1-amino-4- β -hydroxyethylaminoanthraquinone (1 part), 1:4-diaminoanthraquinone (2) and 1:4-di-(β -hydroxyethylamino)-anthraquinone (2) yields strong fast bluish-violet dyeings on cellulose acetate.

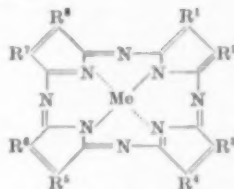
C.O.C.

Blue and Violet Tetra-azaporphin Dyes for Polyesters

ICI

BP 762,478

Dyes of formula—



(Me = metal of atomic number 24–29; R¹ and R² = H, Alk or together form part of an alicyclic ring; R³, R⁴, R⁵, R⁶, R⁷ and R⁸ = H, Alk or any or all of the pairs may be used together to form a subst. or unsubst. alicyclic, aromatic or quinonoid ring) can be applied to polyesters, e.g. by the process of BP 651,694 (J.S.D.C., 67, 287 (1951)) or from solution in a suitable solvent. Thus Terylene (200 parts) powder is mixed with Ni octamethyltetraazaporphin and then intensively dried, melted and spun to yield violet filaments.

C.O.C.

Improving the Rubbing Fastness of Azoic Dyeings and Prints

Ciba

BP 762,510

The soaping of azoic dyeings and prints can be replaced by treatment with the product obtained by treating ≤ 4 mol. of an $\alpha\beta$ -alkyleneoxide with 1 mol. of a compound containing ≤ 1 basic primary or secondary amino group or a basic tertiary group and an alcoholic OH group, or with a salt or quaternary ammonium salt of such a product.

C.O.C.

Dyeing Natural Fibres, Regenerated Cellulose or Cellulose Derivatives, Polyester or Acrylic Fibres

Fontaine Converting Works

BP 760,041

The material is treated at high temperature with a dye in an organic liquid which is miscible with water and which does not evaporate at the temperature of dyeing. Alternatively, the material may be treated with the organic liquid alone and then while at the high temperature and while saturated with the liquid treated with an aqueous solution or dispersion of a dye. The temperature of dyeing is $> 212^\circ\text{F}$. and preferably as near to the softening temperature of the material as possible. Thus thoroughly cleansed and dried polyacrylonitrile fibre is passed through a diethylene glycol solution of a basic dye at 370°F ., the time of immersion being 8–10 sec., surplus liquor squeezed off and the material passed into cold water. The resulting dyeing is much deeper than that obtained by any other method and has excellent fastness to washing and rubbing.

C.O.C.

Increasing the Dye-receptivity and Moisture Absorption of Acrylonitrile Polymers

Chemstrand Corp.

USP 2,726,132

Treatment at $100\text{--}200^\circ\text{C}$. with a half salt of an aromatic sulphonic acid and a compound of formula $\text{X}-(\text{CHR})_n\text{NH}_2$ (R = H or Alk of 1–4 C; X = NH_2 or OH; n = 2 or 3), e.g. the half acid salt of ethylene diamine and benzene sulphonic acid, imparts greatly increased affinity for acid dyes and increased water absorbent properties without affecting the mechanical properties of the fibres.

C.O.C.

Dyeing Acrylonitrile Polymers or Copolymers

CFM

BP 762,044

Ferrous compounds, e.g. FeSO_4 or ferrous ammonium sulphate, are excellent reducing agents for use with copper compounds in the cuprous ion method of dyeing acrylonitrile fibres. They are best used together with an agent with which the residual iron compound forms a soluble complex, e.g. a polyphosphate.

C.O.C.

Improving the Wet Fastness of Direct Dyes on Cellulose

FBy

BP 761,668

The increase in wet fastness obtained by treating the dyeings with quaternary ammonium compounds of high mol.wt. and free from ether groups, especially quaternised peralkylation products of polyamines, is considerably improved if a water-soluble sulphate is present in the bath.

C.O.C.

Dyeing Hare and Rabbit Fur Felts with Heavy Metal Complexes of Dyes

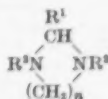
Gy BP 762,049
Half and fully planked fur felts are evenly and thoroughly dyed with the heavy metal complexes of azo and methin dyes (2 mol. dye : 1 atom metal) containing no acid water-solubilising groups except carboxyl groups bound in complex linkage if dyeing is done in a well circulating liquor at pH 3-5. Usually pre-neutralisation of the felts is unnecessary and good exhaustion is obtained.

C.O.C.

1:3-Diazacycloalkyl Compounds — Gas Fume Fading Inhibitors

Celanese Corp. of America
Compounds of formula—

BP 760,335



($\text{R}^1 = \text{H}$, Alk or Ar; $\text{R}^2 = \text{cycloalkyl}$, aralkyl or Ar; $n > 1$), are obtained as concentrated aqueous dispersions by treating a compound of formula R^1CHO with one of formula $\text{R}^2\text{NH}(\text{CH}_2)_n\text{NHR}^3$ in an aqueous solution of a dispersing agent. These dispersions or pastes are extremely stable to storage. They are used in disperse dyebaths as gas fume fading inhibitors, 1:3-diphenylimidazolidine being particularly suitable for this purpose.

C.O.C.

Improving the Dyeing Properties of Synthetic Thermoplastic Material

Raduner & Co.

BP 758,745

The dyeing properties of acrylonitrile polymers and copolymers, nylon or Terylene are improved by treatment with strong bases at elevated temperatures in absence of water. Thus polyacrylonitrile fabric treated for 2 min. in a 10% soln. of NaOH in ethylene glycol at 150°C. and then thoroughly washed in warm water was readily dyed deep brown with Chlorantine Fast Brown BRLL (Ciba) whereas untreated it is only slightly stained.

C.O.C.

Print-like Effects on Textiles and Films

Bradford Dyers' Assocn.

BP 759,595

Material containing a nitrogenous synthetic polymer is subjected in selected areas to mechanical pressure, e.g. by calendaring, preferably in presence of heat and moisture. It is then treated before or during dyeing with an anionic dye with a resist agent which reduces the affinity of the material for the dye. The areas which have been subjected to mechanical pressure are less affected by the resist agent and so dye more deeply than the other parts of the material.

BP 759,625

The process is applicable to natural proteinaceous materials with the exceptions of silk and regenerated proteinaceous material.

C.O.C.

Colouring Glass Fabric with a Resinous Coating dyed with an Anthraquinonoid Vat Dye

General Aniline

USP 2,722,488

The fabric is coated with the precondensate of a water-soluble, resinous polyhydroxy compound, preferably vinyl alcohol, with a water-soluble interpolymers of maleic anhydride and vinyl methyl ether, vinyl ethyl ether or vinyl acetate. The dye may be applied in keto form together with the precondensate or it may be applied after the cured resin has been formed on the fabric.

USP 2,722,489

The dye is applied in the anthrol or vat acid form.

C.O.C.

Dyeing and Printing

S.A.I.C.A. Societe Anonima Italiana Colori Affini

BP 759,293

The process of BP 733,099 (J.S.D.C., 71, 562 (1955)) is applicable to any class of dye having affinity for the fibre.

C.O.C.

Technology of Modern Oxidation Hair Dyes—II (IV p. 63)

Cellulose Triacetate (VI p. 71)

Supertwisting and Dyeing Polyamide and Polyester Filament Yarns (VI p. 71)

IX—PRINTING

PATENTS

Screen Printing Machine

VEB Textilmaschinenbau Zittau

BP 763,030

The cloth passes successively below several screens. An endless band running at the side of the printing blanket carries pattern repeat stops which can be finely adjusted. This band sets the screen carriages in correct registration and stops the printing blanket.

C.O.C.

Improving the Fastness to Rubbing of Pigment-printed Textiles

General Electric Co.

BP 758,735

Textiles which have been printed with a paste made up of a pigment, a resinous or cellulose derivative carrying agent and a binder are irradiated with high energy electrons. This converts the binder into a product of higher molecular weight.

C.O.C.

Flame-resistant Finish for Cellulosic Materials

Dow Chemical Co.

USP 2,725,311

The material is impregnated with a bis-(ω -monohalogenoalkyl)- ω -monohalogenoalkane phosphonate in a volatile solvent, e.g. with bis-(2-bromoethyl)-2-bromoethane phosphonate and bis-(2-chloroethyl)-2-chloroethane phosphonate. They are produced by adding a phosphorus trihalide to an alkylene oxide in a solvent and heating to $< 150^\circ\text{C}$. The yields are almost quantitative and the resulting solutions may be used directly for treating cellulosic textiles or paper.

C.O.C.

Dry Release Transfers

Meyercood Co.

BP 759,102

A dry release coating is applied to a temporary carrier. On this coating there is applied a layer of a resinous film-forming material readily separated by dry release from the first coating. Subsequently there are separately applied several lacquer layers one at least of which is in the form of a design. Each lacquer layer contains a resinous-film forming ingredient and a selective solvent for it, the resinous material in each layer differing from the one in the previous layer so that the solvent in each lacquer layer has no effect on the resinous material in the previous layer.

C.O.C.

Light-sensitive Sheets for Producing Planographic Prints

Chemische Fabrik L. van der Grinten

BP 761,493

Light-sensitive Sheets for producing Graphic Prints

Chemische Fabrik L. van der Grinten

BP 761,494

Colour Photography

Gevaert Photo-Produkten and Chromoact Film Industries

BP 762,751

A development of the method of producing apparently trichromatic images from bicoloured negatives described in BP 710,481 (J.S.D.C., 70, 375 (1954)).

C.O.C.

Light-sensitive Sheets for producing Transfer Images and Graphic Prints

Chemische Fabrik L. Van der Grinten

BP 761,495

Colour Separation Negatives for Four-colour Reproduction

Time

BP 761,535

Diazotype Material

General Aniline

BP 762,140

In sensitising emulsions containing silica use as binder of a water-soluble or water-dispersible starch or water-soluble polymer of a compound containing a $\text{CH}_2\text{CH=}$ or $\text{CH}_2\text{C}(\text{CH}_3)=$ group enables rapid printing to be economically achieved together with images of higher density on a clean white ground.

C.O.C.

Diazotype Compositions containing Silica

General Aniline

BP 762,117

Modification of BP 647,617 (J.S.D.C., 67, 167 (1951)). Use of iron-free silica of particle size 0.015-0.20 μ , e.g. Aerosil (G. L. Cabot) or Santocel (Monsanto), gives much superior results without need for a special binding agent for the silica particles.

C.O.C.

Fixing Xerographic Prints

International Business Machines Corp.

USP 2,726,166

Dyeing or Printing Cellulose with Diazo or Polyazo Dyes containing Copper bound to α -Hydroxyazo Groups (VIII p. 73)

Improving the Rubbing Fastness of Azoic Dyeings and Prints (VIII p. 73)
 Dyeing and Printing (VIII p. 74)
 Decorated Flexible Plastic Sheet (XIII p. 78)

X—SIZING AND FINISHING

Drying of Sized Fibres

K. Ramaszedér

Textil Praxis, 11, 991-998 (Oct. 1956)

A detailed investigation into the behaviour of sizes on drying and the drying of sized yarns under varying conditions. B.K.

Sizing of Cellulose Acetate Filament Yarn—Relation of the Apparent Stiffness of Sized Warp to the "Chika" in Cellulose Acetate Continuous-filament Fabrics

M. Iino and H. Torigata

J. Soc. Textile Cellulose Ind. Japan, 12, 665-670 (Sept. 1956)

The relations of the apparent stiffness of sized warp to "Chika" defects in cellulose acetate filament fabrics were studied and the following results were obtained—(1) It is found that the "Chika" defects are caused by the highly stiffened parts of the sized warp and that the "Chika" could be prevented by limiting the stiffness of the sized warp. (2) The stiffness of the sized yarn depends on the magnitude of the size pick-up and the concentration of the size solution. "Chika" seems to be the same phenomenon as "Tear" described in *Fabric Defects* (McGraw-Hill Book Co.).

AUTHORS

Use of Synthetic Detergents in the Finishing of Fabrics containing Wool

R. C. Cheotham and C. S. Whowell

World Congress on Surface-active Agents, 2, 438-441 (1954)

The ways in which synthetic detergents can advantageously replace soap in the scouring, milling, and raising of wool and wool-protein rayon mixture fabrics are described. W.K.R.

Reaction of Formaldehyde with Cellulosic Fibres

H. K. Woo, J. H. Dusenbury, and J. H. Dillon

I—Rate and Mechanism of the Reaction

Text. Research J., 26, 745-760 (Oct. 1956)

The acid-catalysed reaction of formaldehyde with cotton, Fortisan, and viscose rayon has been investigated under conditions comparable with those used in finishing. The variables studied were pH (2.0, 2.2, and 2.4), formaldehyde concentration, temperature, and reaction time. At pH 2.2 and 2.4 the rate-controlling process appears to be the formation of methylene-ether cross-linkages in the amorphous regions of the fibre. The activation energy of this reaction is 13.4-17.7 kcal./mole. At pH 2.0 the reaction in the amorphous regions is very rapid, and the rate-determining step is a diffusion-controlled reaction in the crystalline regions with an activation energy of 3.4-6.3 kcal./mole. This may be due to the use of a different buffer system for the pH-2.0 experiments (HCl-KCl as against HCl-K H phthalate). Both rate and extent of reaction appear to depend on the state of internal order of the material, being least for cotton, the material of highest crystallinity. X-Ray diffraction studies of the treated fibres gave results consistent with the proposed reaction mechanisms.

II—Mechanical Behaviour

Ibid., 761-783

The mechanical properties of formaldehyde-treated samples were compared with those of three sets of control samples—untreated fabric, fabric treated with distilled water under the experimental conditions, and the fabric treated under the same conditions with the acid solution used in the formaldehyde treatment. Cotton showed a deterioration in mechanical properties on formaldehyde treatment compared with the acid-treated control, whilst Fortisan and viscose rayon were improved by such treatment. The deterioration in mechanical properties caused by acid alone was most marked in the case of Fortisan. Improvement in crease resistance on formaldehyde treatment was greatest for cotton and least for viscose rayon. Marked improvement in crease recovery was obtained only with those fabrics treated at pH 2.0, indicating that a high degree of cross-linking in

the amorphous regions of the fibre is necessary for high crease resistance. J.C.F.

Effect of Yarn Twist on Fabric Crease Recovery

R. Steele

Text. Research J., 26, 739-744 (Oct. 1956)

Certain cellulosic fabrics have been shown to exhibit anisotropic creasing behaviour. This is attributed to interaction between yarn twist and the torsional deformation of individual yarns produced when the fabric is creased in directions at an angle to both warp and weft. Treatment of such fabrics with dimethylolurea improves the crease resistance and also makes it more nearly isotropic. Fabrics made from fibres of greater intrinsic resilience, such as wool, nylon, and cellulose acetate, show essentially isotropic creasing properties. J.C.F.

Studies on Water Repellency of Organic Silicon Compounds. III—Water Resistance of Fabrics treated with Methylpolysiloxane containing Thiuronium Chloride

K. Nakao, N. Wada, K. Nakasima, and S. Nozakura

Text. Research J., 26, 784-788 (Oct. 1956)

A linear methylpolysiloxane containing thiuronium groups has been prepared by the reaction of chloromethylmethylpolysiloxane (D.P. 17) with thiourea in alcohol. Application of the product from aqueous solution, followed by treatment with ammonia, heat-curing, or ageing, imparted a durable water-repellent finish to silk and cotton fabrics. J.C.F.

Acrylic Fibres

XXI—Influence of Aftertreatment on the Fibre Properties

M. Katayama

J. Soc. Textile Cellulose Ind. Japan, 12, 621-626

(Sept. 1956)

The influence of aftertreatment (cold drawing, heat stretching, and heat treatment) on the mechanical properties of polyacrylonitrile fibres was studied, and the following results were obtained—(1) Young's modulus and the rigidity change at the second transition temperature; (2) desirable heat-treatment is steaming in a fully relaxed state.

XXII—Crimp of Polyacrylonitrile Fibres

M. Katayama and Z. Saito

Ibid., 626-631

The gear crimp and natural crimp methods were studied, and the following results were obtained—(1) the suitable temperature for gear crimp method of polyacrylonitrile fibres is 140°C., and of acrylamide ($\text{CH}_2\text{CH}(\text{CO}-\text{NH}_2)$) copolymer fibre is 130°C.; (2) natural crimp is possible as viscose rayon fibre. AUTHORS

PATENTS

Fixing Mechanical Finishes

Bradford Dyers' Assocn.

BP 761,548

The material is impregnated with a fixing agent and then while saturated given a mechanical finish after which it is dried and if needed baked to set the fixing agent. Thus mercerised cotton poplin is impregnated with a solution of dimethylol urea containing a little ammonium dihydrogen phosphate and stearanido-methyl pyridinium chloride. It is then crepe embossed, dried and baked for 3 min. at 150°C. The resultant crepe embossed finish is resistant to repeated washing. C.O.C.

Flame-resistant Finish

Victor Chemical Works

BP 759,329

A flame-resistant finish fast to washing is obtained without affecting the physical qualities and texture of the material by treating it with a polymer or partly brominated polymer of a di- β -alkyl- β -cyano ethane phosphonate and then curing at moderate temperature. C.O.C.

Extensible Fabrics

Bleachers' Assocn. Ltd.

BP 758,639

An extensible fabric, i.e. one which when stretched warps does not return to its original length when the tension is released, is produced by treating a fabric containing non-thermoplastic fibres in a compressive shrinking machine so that its length is shorter than it is normally. The shrunk fabric is then treated with varnish, e.g. a linseed oil varnish, and then baked, e.g. at 350°F. C.O.C.

cellulose (prepared by hydrolysis), the density of amorphous cellulose is calculated. The value obtained (1.488) is much lower than that assumed by previous investigators, and when this value is used in the calculation of A.F. values by the density method, the results, though higher than the ethanolysis values, are much lower than those obtained by the use of previously accepted values of the density of amorphous cellulose. A.E.S.

Sorption and Desorption Kinetics of Water in a Regenerated Cellulose

A. C. Newna

Trans. Faraday Soc., **52**, 1533-1545 (Nov. 1956)
Characteristics of sorption and desorption kinetics on the lower limb of the hysteresis loop of a water-cellulose system have been determined. The kinetics may be interpreted in terms of a coupled diffusion-relaxation mechanism, which should explain the characteristics of the non-steady-state transfer of vapours in polymers below their glass temperatures. Results suggest that the free energy increase during sorption may be separated into the sum of an increase in free energy due to swelling against unrelaxed internal forces and an increase due to mixing with these forces relaxed. W.R.M.

Interaction of Trifluoroacetic Acid with Cellulose and Related Compounds

A. L. Geddes

J. Polymer Sci., **22**, 31-35 (Oct. 1956)
The reaction of cellulose, cellobiose, and their acetyl derivatives with trifluoroacetic acid has been studied by infrared methods. Cellulose is dissolved by an initial process of swelling, rupture of hydrogen bonds, and micellar dispersion of the cellulose structure. Esterification occurs, possibly by a mechanism involving an intermediate compound. Cellobiose reacts in a similar manner. Partially acetylated cellulose is further acetylated, and the results suggest more rapid esterification of primary than of secondary hydroxyls. W.R.M.

System for Classification of Structurally Related Carbohydrates

H. S. Isbell

Bur. Stand. J. Res., **57**, 171-178 (Sept. 1956);
Research Paper 2707

PATENT

Hydrolysing Cellulose Acetate Films

Cinotechnik

BP 761,791

The film is hydrolysed with caustic soda lye containing an ethyleneoxide-alkylphenol condensate. Hydrolysis is uniform both on the surface and in depth. C.O.C.

Ornamental Effects on Thermoplastic Sheets or Webs

(X p. 76)

Accelerating the Condensation of Organopolysiloxanes

(X p. 76)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Surface-active Agents in the Leather Industry

D. Burton and G. F. Robertshaw

World Congress on Surface-active Agents, **2**, 547-550 (1954)

Brief descriptions are given of the processes used in making different kinds of leather, and the functions of surface-active agents in this industry are discussed. W.K.R.

Interaction of Detergents with Collagen and Leather

K. G. A. Parkhurst

World Congress on Surface-active Agents, **2**, 551-555 (1954)

The equilibrium adsorption of different types of detergent on collagen and leather has been studied by measuring the alteration in shrinkage temperature after treatment. With collagen, the anionic agent lowered the shrinkage temperature considerably, whilst the cationic reduced the shrinkage temperature to a much smaller extent, and the non-ionic had no effect. Qualitatively similar effects, but of smaller magnitude, were observed with chrome-tanned leather. With vegetable-tanned leather, however, the anionic had little effect, but the cationic and the non-ionic (which are known to form stable complexes with vegetable tannins) caused appreciable reduction in

shrinkage temperature. Explanations are given of the mechanisms by which these different effects are produced. W.K.R.

Use of Surface-active Materials as Leather-dyeing Assistants

D. H. Tuck

World Congress on Surface-active Agents, **2**, 564-565 (1954)

Dyeing of leather is limited to 40-70°C. and pH 4-8. The addition of surface-active agents to the dyebath produces brighter and cleaner dyeings, but foaming can be troublesome and should be controlled by the use of a silicone anti-foaming agent. With anionic dyes, anionic and non-ionic auxiliaries give improved levelness and penetration, whilst cationics give reduced penetration, full surface colour, and better exhaustion owing to co-precipitation and the formation of a lake on the surface of the leather. Cationics are sometimes added at the middle or end of dyeing to cause laking of the dye. With cationic dyes, better penetration and levelness are obtained by the addition of a cationic surface-active agent. A brief description is given of the theory of leather dyeing. W.K.R.

Synthesis of Cystinol

J. C. Crawhall, D. F. Elliott, and K. C. Hooper

J.C.S., 4066-4068 (Nov. 1956)

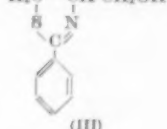
Cystinol (II) and some deriv. of cysteinol (I) are prepared by the reduction of the relevant 4-alkoxy-carbonyl-2-phenylthiazoline with LiAlH_4 . The thiazoline alcohol (III) so obtained yielded on hydrolysis a soln. which presumably contained I, although it could not be isolated pure; mild oxidation of the soln., however, afforded II as a cryst. dihydrochloride. The conditions are also investigated under which cysteinol or cystinol might be isolated from the mixture obtained when a peptide containing C-terminal cysteine had been reduced by a metal hydride and hydrolysed. S-Benzyleysteinol, a cpd. of possible value for the isolation of cysteinol from hydrolysates, has been prepared from S-benzyleysteinine methyl ester and isolated as the oxalate.



(I)



(II)



(III)

PATENTS

H.H.H.

Tanning

Boehme Fettchemie

BP 758,674

High grade leathers are obtained if the skins are first treated with an aldehyde-nitrogenous compound pre-condensate and then with either a vegetable or synthetic tanning agent or with sulphite waste liquor. Use of this process at least halves the time needed for tanning and also reduces the amount of tanning agent needed. C.O.C.

Modified Aminoplast Resin Complexes—Tanning Agents

L. Sellot

BP 762,774

Diacydiamide is heated with a water-soluble mono- or polycarboxylic acid, e.g. HCOOH , the resulting product condensed with HCHO and then treated with a polyvalent metal salt, e.g. Al acetate. This yields tanning agents which give leather having high shrinkage temperature and resistance to water as well as fullness and fastness to light. C.O.C.

Retanning Agents

FBy

BP 761,982

The salts of condensates of sulphones of monohydric phenols with primary or secondary non-aromatic amines and formaldehyde are excellent re-tanning agents although they do not precipitate gelatin and their tanning effect on skins prepared for tanning is poor.

Amphoteric condensates obtained from a monohydric phenol, a primary or secondary amine and an aldehyde or ketone, are excellent agents for the re-tanning of leather and produce effects as good as those obtained by the above process.

Fat-liquoring DuP

A dry-cleanable leather which if dyed does not bleed into water or solvent is obtained by using as the fat-liquor on leather of pH ≥ 5 an aqueous solution of a water-soluble Werner-type complex in which trivalent nuclear Cr atoms are condensed with carboxylic acid groups of > 9 C, e.g. stearatochromic chloride.

C.O.C.

Effects of X-Rays on Dilute Aqueous Solutions of Amino Acids [Formation of Melanin] (IV p. 65)

XIII—RUBBER; RESINS; PLASTICS

Molecular Weights in Acrylonitrile Polymerisation P. F. Onyon

J. Polymer Sci., **22**, 19–23 (Oct. 1956)

Rates of polymerisation of acrylonitrile have been measured, and also the intrinsic viscosities of the polymers formed. Ratios are derived for kinetic constants. Results are consistent with occurrence of radical termination by disproportionation.

W.R.M.

Polyacrylonitrile: Molecular Weight-Viscosity Relation P. F. Onyon

J. Polymer Sci., **22**, 13–18 (Oct. 1956)

Number-average molecular weights \bar{M}_n (28,000–1,000,000) have been determined by osmometry and from intrinsic viscosities $[\eta]$ measured in dimethylformamide at 25°C. The equation—

$$[\eta] = 3.92 \times 10^{-4} \bar{M}_n^{0.76}$$

is obeyed within the limits of experimental error.

W.R.M.

Copolymers of Allyl Alcohol and Acrylonitrile produced by Dye-sensitised Photopolymerisation G. Oster and Y. Mizutani

J. Polymer Sci., **22**, 173–178 (Oct. 1956)

Copolymers of relatively high molecular weight have been produced using acriflavine as a sensitizer for photopolymerisation.

W.R.M.

Thermal Degradation of Polymethacrylonitrile. I—Separation of Coloration and Depolymerisation Reactions

N. Grassie and I. C. McNeill

J.C.S., 3929–3933 (Oct. 1956)

Some preliminary experiments are described to define the conditions under which depolymerisation and coloration reactions occur and to show how they can be separated for individual study. It is found that the colour which develops in polymethacrylonitrile at 120°C. is due to a reaction initiated at impurities in the polymer chains. These are eliminated by suitable purification of the monomer and by carrying out the polymerisation in a vacuum in presence of *anti*-azoisobutyronitrile as catalyst. By using such polymers, the depolymerisation reaction, which occurs at 220°C. with quant. yield of monomer, can be investigated in absence of complications due to coloration. The coloration is most conveniently studied in copolymers of methacrylonitrile with traces of methacrylic acid, which act as centres of initiation.

H.H.H.

Chemical Activity of γ -Irradiated Polymethyl Methacrylate

L. A. Wall and D. W. Brown

Bur. Stand. J. Res., **57**, 131–136 (Sept. 1956);
Research Paper 2702

Studies of the polymerisation and depolymerisation of γ -irradiated polymethyl methacrylate suggest the presence of long-lived free radicals in the solid polymer. With a dose of 10^7 roentgens the free-radical concentration is estimated to be at least 10^{-4} m. When irradiated in air the polymer contains peroxide structures, the decomposition of which is accelerated by *tert*-butylcatechol and is associated with scissions in the polymer chains.

W.R.M.

PATENTS

Coloured Polyphosphate-Dye Polymers ICI

BP 760,019

The products obtained by reacting a solubilised potassium polyphosphate with an aqueous solution of a dye containing isothiuronium salt groups are used in the formulation of non-inflammable moulding compositions and as additives to lubricating oils. Thus K polyphosphate (2.5 g.) was added to brine (50 ml.) of sufficient strength to give K:Na::1:2. The mixture was stirred for 1 hr., the solution poured off and water (180 ml.) added. After rapid agitation nearly all the swollen residue dissolved. The solution was filtered. To the clear liquor (100 ml.) was slowly added, with constant stirring, a 1.5% soln. (270 ml.) of the blue dye obtained by treating Cu tri-(chloromethyl)phthalocyanine with tetramethyl thiourea. A flocculent blue precipitate was formed which on washing and drying yielded a dark blue crumbly solid which did not soften at 250°C.

C.O.C.

Decorated Flexible Plastic Sheet United States Rubber Co.

BP 761,833

A thin layer of resinous film-forming material is printed on an inextensible carrier sheet, e.g. of paper, and dried. A design is then printed on this layer and dried. The main body of the resinous film-forming material is then printed on and the whole fused and stripped off the carrier.

C.O.C.

Coloured Articles from Plastic Material Reinforced with Textile Fibres

M. P. de Vinzelles

BP 762,052

A layer of resin containing a dye or pigment is deposited in or on a mould followed by the main charge of resin-impregnation fibres. The process is particularly suitable for making composite articles from glass fibres and polyester resins.

C.O.C.

Preventing Discoloration of White or Light-coloured Paints on Rubber (V p. 70)

Ornamental Effects on Thermoplastic Sheets or Webs (X p. 76)

Accelerating the Condensation of Organopolysiloxanes (X p. 76)

XIV—ANALYSIS; TESTING; APPARATUS

Complexometric Estimation of Sulphate

B. Philipp and H. Hoyme

Faserforsch. und Textiltech., **7**, 525–527 (Nov. 1956)

A method is described for the estimation of sulphate in viscose spinning baths using a phthalein-potassium complex. Standard errors are said to be less than with other methods.

W.R.M.

Determination of Microquantities of Sulphate Ion derived from Small Samples of Textile Material

J. M. Bather

J. Textile Inst., **47**, T 567–T 569 (Nov. 1956)

A modification of the filtering technique of Belcher, Gibbons, and West's method (*Chem. and Ind.*, 127 (1954)) for estimating small quantities of sulphate ion. An accuracy of 10% is obtained with 0.1–2.0 mg. of sulphate.

P.T.S.

Colorimetric Determination of Phosphorus in Microgram Quantities

N. S. Ging

Anal. Chem., **28**, 1330–1333 (Aug. 1956)

Orthophosphate can be determined in presence of readily hydrolysed organic and condensed phosphates by a modified molybdenum blue method in which a low concn. of acid is used. Stable and labile phosphates can be determined separately.

L.T.W.

Gradient-elution Chromatography of Phosphates

J. A. Grande and J. Beukenkamp

Anal. Chem., **28**, 1497–1498 (Sept. 1956)

By employing gradient, rather than discontinuous, elution, the ion-exchange chromatographic separation of phosphates from mixtures is improved and the elution time shortened.

L.T.W.

General Determination of Acetyl

A. Chaney and M. L. Wolfson

Anal. Chem., **28**, 1614–1615 (Oct. 1956)

An apparatus is described by which accurate values of both O- and N-acetyl may be obtained with a minimum of

attention by the operator. The method is a modification of the Ost acid-distillation method and is applicable to wettable carbohydrates and other organic compounds.

L.T.W.

Spectrophotometric Determination of Microgram Quantities of Disodium Dihydrogen (Ethylene-dinitrilo)tetra-acetate

O. Menis, H. P. House, and I. B. Rubin

Anal. Chem., **28**, 1439-1441 (Sept. 1956)

The absorbance of a soln. of the cupric complex of (ethylenedinitrilo)tetra-acetic acid (EDTA) in a phosphate buffer at pH 11 is measured at 250 m μ , the complex being obtained by adding an excess of cupric sulphate to the test soln. Cupric sulphate in an identical buffer is used as reference soln. Good accuracy is obtained with samples containing 125-1500 μ g. of EDTA. Nickel, cobalt, and chromate ions interfere.

L.T.W.

Analysis of Mixtures of D-Glucose and D-Mannose by Paper Electrophoresis

D. R. Briggs, E. F. Garner, R. Montgomery, and F. Smith

Anal. Chem., **28**, 1333-1335 (Aug. 1956)

In a simple method for the analysis of mixtures of glucose and mannose in which there is a high proportion of glucose, separation is effected by paper electrophoresis. The sugars are then determined by the phenol-sulphuric acid method. The construction of an inexpensive paper-electrophoresis apparatus is described.

L.T.W.

Quantitative Paper Chromatography of Methylated Aldose Sugars

W. C. Schaeffer and J. W. van Cleave

Anal. Chem., **28**, 1290-1293 (Aug. 1956)

An improved method is outlined for the quantitative determination of methylated aldose sugars after their separation from mixtures by paper chromatography. The method is a modification of a colorimetric procedure using aniline hydrogen phthalate as chromogenic agent (Blas, Machobouf, and Nunez, *Bull. Soc. Chim. biol.*, **32**, 130 (1950)), the reaction being carried out in tetraethylene glycol dimethyl ether. This solvent ensures homogeneity and provides for high sensitivity and good reproducibility. An examination of the behaviour of the methyl ethers of D-glucose, D-mannose, D-galactose, and D-xylose indicates the general applicability of the method.

L.T.W.

Preparation of Potato Amylose for Quantitative Colorimetric Iodimetry

J. L. Lambert and S. C. Rhoads

Anal. Chem., **28**, 1629-1630 (Oct. 1956)

Pure potato amylose is obtained by suspending raw potato starch in water for 2 hr. at 57-60°C., reprecipitating the extracted material three times with n-pentyl alcohol, dehydrating it by successive treatments with methanol and n-pentyl alcohol, and finally drying it at room temperature.

L.T.W.

Analysis and Identification of Aliphatic Alcohols—Applications to Complex Synthetic Products

R. Wemelle, L. Bourgeois, and J. Bolle

World Congress on Surface-active Agents, **1**, 224-227 (1954)

Difficulties in the Analysis of Commercial Detergent Products

C. Paquet

World Congress on Surface-active Agents, **1**, 171-174 (1954)

Chromatography of Dye Intermediates. V—Naphthylamine- and Naphthol-sulphonic Acids

J. Latinák and L. Skalický

Chem. Listy, **50**, 1598-1601 (1956)*Chem. Abs.*, **50**, 16258 (25 Nov. 1956)

On non-impregnated paper the systems propyl alcohol-aq. NaHCO₃ (2:1) and butanol-pyridine-water (3:1:1) give similar separation of 21 naphthylamine- and 17 naphthol-sulphonic acids. On paper impregnated with 5% aq. NaHCO₃ improved separation of all the naphthol-sulphonic acids is obtained.

C.O.C.

Separation of Some Disperse Dyes by Paper Chromatography

K. Elliott and L. A. Telesz

J.S.D.C., **73**, 8-11 (Jan. 1957)

A method is described for the qualitative analysis of some disperse dyes on paper using petroleum ether (b.p. 65-70°C.) saturated with 98% methyl alcohol (2% water by volume). The appropriate amount of dye solution is spotted on the paper. After exposure to an atmosphere

saturated with eluant vapour, the chromatograms are run overnight at 19-21°C. Details of the method and some *R_f* values are given in the text.

AUTHORS

Determination of the Relative Tinting Strength of Carbon Black (in Printing Ink)

A. Voet

Am. Ink Maker, **34** (5), 63 (1956)*Chem. Abs.*, **50**, 16130 (10 Nov. 1956)

Weighed portions of sample and standard blacks are milled into a white printing ink on an automatic muller. Tinting strength is determined photoelectrically. Accuracy is about $\pm 1\%$.

C.O.C.

Air Permeability of Surfaces

J. Dechant

Faserforsch. und Textiltech., **7**, 510-515 (Nov. 1956)

An apparatus is described for the measurement of air permeability of textile materials. The flow of air through fabric is considered theoretically, and experimental results are compared with theoretical predictions.

W.R.M.

Features of Cellulose Fibre Structure shown by Differential Dyeing

C. F. Goldthwait and W. G. Sloan

Amer. Dyestuff Rep., **45**, 813-819 (5 Nov. 1956)

Cotton is dyed with a mixture of Chlorantine Fast Green BLL (Ciba) (mol. wt. 1350) and Diphenyl Fast Red 5BL Supra I (Gy) (straight chain, mol. wt. 675). Differences in structure are shown qualitatively by differential absorption of dye after stripping for 30 sec. in boiling water. The relative amount of each dye absorbed depends on "openness of structure", "wall thickness", "effectiveness of contact of dye molecules with adsorption sites of fibres for direct dyes in a dyebath", and thus on the maturity and the degree of drying of the fibres. Qualitative results so obtained can be applied in investigations of the structure of ramie, rayons, and modified cottons. References are made to theoretical work on dye affinities.

P.T.S.

Swelling of Cotton Fibres in 15% Sodium Hydroxide as a Microscopic Test for Chemical Damage

P. A. Koch and H. Hefti

Textil-Rund., **11**, 512-519 (Sept.), 645-655 (Nov. 1956)

Good correlation exists between the D.P. and the type of swelling obtained, independently of the method of chemical damage. The "mushroom head" reaction can be applied at any stage of processing, since the usual operations are normally without influence on the test.

S.R.C.

Identification and Estimation of Fibres in Unions

Mlle Charles and M. Salé

Rayonne, **12**, 1569-1592 (Nov. 1956)

Qualitative and quantitative analysis of fibres in unions by means of physical, microscopical, and chemical tests.

B.K.

Simple Method of Identifying Man-made Fibres

J. Khachoyan

Teinter, **21**, 863-871 (Nov. 1956)

Two differential stains known as Identex No. 1 and No. 2 are used in combination with a number of chemical tests and solubilities. Colour plates showing the stains and a table of chemical reactions are given.

S.R.C.

Isolation of the Skin of Viscose Rayon Fibres

R. Maré

Faserforsch. und Textiltech., **7**, 493-496 (Nov. 1956)

Slattery's method of isolation is criticised. A new method is described, consisting in swelling transverse sections of viscose rayon in Marshall's solution. The method is similar to that developed by Schwertasek for the separation of skin from the core of polyamide fibres.

W.R.M.

Identification of Synthetic Fibres by Micro Fusion Methods

D. G. Grabar and R. Haensly

Anal. Chem., **28**, 1586-1589 (Oct. 1956)

Synthetic fibres are identified by observation of their behaviour on heating alone and in admixture with *p*-nitrophenol, the melting point being determined in each instance. The observations are carried out with a hot-stage polarising microscope; a silicone oil is used to exclude air from the fibres during heating and to improve the image. Micro fusion data for thirteen synthetic fibres are tabulated.

L.T.W.

Identification of Inorganic Fibres

W. Boboth and U. Müller

Faserforsch. und Textiltech., **7**, 497-504 (Nov. 1956)
 Microchemical methods are described and a scheme is given for the identification of inorganic fibres. W.R.M.

Microscopical Examination of Dyeing and Finishing Processes

H. Roumuth

Z. ges. Textilind., **58**, 774-783 (5 Oct.),
 913-925 (5 Dec. 1956)

The use of the microscope in following a wide range of dyeing and finishing processes is described. A number of illustrations in both colour and black-and-white are included. B.K.

Survey of American Colour Specifications— 1955

Inter-Society Colour Council

Off. Dig. Fed. Paint Varn. Prod. Clubs,
28, 902-921 (Oct. 1956)

A report by a subcommittee of the Problems Committee of the Inter-Society Colour Council. The various sections take the following broad form: I—*Systems of Colour Specification* gives the terms, types of notation, references, and other details for the Munsell and Ostwald systems. Seven other prominent systems are referred to by title. II—*Reflecting Colour Standards* is divided into two sections: one for those giving relatively complete coverage of colour space, and the other for those with abridged or regional coverage. III—*Transmitting Standards* lists 14 such sets. IV lists the names of *Instruments for Colour Measurement* with a key to the type of measurement for which they are intended. V gives the classification of all standards and methods according to material: the sub-headings include—ceramics and plastics, dyes, oils and fats, paint finishes, paper, pigments, and textiles. VI—*Colour Codes* gives references for codes used in eight fields, including those of gas cylinders and electrical insulation. VII is an appendix; it gives detailed references for all the material in the foregoing sections, and also furnishes a short list of abbreviations. J.W.D.

Current Developments in Colour Fastness Testing in the United Kingdom

P. W. Cunliffe

Amer. Dyestuff Rep., **45**, P 791-P 793 (22 Oct. 1956)

Current Developments in Colour Fastness Testing in the U.S.A.

C. A. Sylvester

Amer. Dyestuff Rep., **45**, P 719-P 721, P 730 (24 Sept. 1956)

Current Developments in Colour Fastness Testing in France

J. P. Niederhauser

Amer. Dyestuff Rep., **45**, P 794 (22 Oct. 1956)

Current Developments in Colour Fastness Testing in Switzerland

H. Ris

Amer. Dyestuff Rep., **45**, P 795-P 796 (22 Oct. 1956)

Xenon Fading Lamp XBF 6000

H. Ilzhöfer

Textil Praxis, **11**, 1220-1225 (Dec. 1956)

A detailed description together with spectral curves of the xenon lamp XBF 6000, which is a water-cooled apparatus for light-fastness exposure tests. It is claimed that by use of a suitable filter the ultraviolet and visible radiation passing through the apparatus give a continuous spectrum similar to that of daylight. Results obtained with a wide range of patterns as well as the standard blue wool scale gave good agreement with daylight-exposure tests. B.K.

Comparison of Methods for Testing the Fastness to Gas-fume Fading of Dyes on Cellulose Acetate

P. Rabe and R. Dietrich

Melliand Textilber., **37**, 321-324 (March 1956);

Amer. Dyestuff Rep., **45**, 737-740 (24 Sept. 1956)

Gas fading is caused by oxides of nitrogen. Apparatus to cause and measure gas fading is critically examined and a comparison is made with normal gas fading of fabrics in use. P.T.S.

Colour Test for Thiols and Thiol Esters

R. Benesch, R. E. Benesch, M. Gutcho, and L. Laufer

Science, **123**, 981 (1956);

J. Amer. Leather Chem. Assoc., **51**, 621 (Nov. 1956)

The reaction products between *N*-ethylmaleimide

(NEM) and thiols are made alkaline to develop a red colour. The colour reaction provides an excellent means for making visible such thiol compounds as cysteine in paper chromatograms. The paper is air-dried, dipped in 0.05 M-NEM in absolute isopropanol, air-dried for 15 min., and then dipped in 0.25 M-KOH in absolute propanol. Pink to red spots develop where the thiol is present. C.J.W.H.

Improvement of the Hydrazine Method for Determination of C-Terminal Amino Acids

J. H. Bradbury

Nature, **178**, 912-913 (27 Oct. 1956)

Peptides or amino acids are heated with hydrazine sulphate as catalyst and hydrazine in a sealed tube at 60°C. for 16 hr. This results in a better yield of amino acids than the method of Atabari, which involves treatment with hydrazine at 100°C. for about 10 hr. W.R.M.

Chemical Methods for Evaluating Carroted Fur

H. G. Fröhlich

Z. ges. Textilind., **58**, 947-958 (20 Dec. 1956)

A detailed investigation of methods of estimating the degree of carrotting of hare fur and wild and tame rabbit furs using alkali and acid solubility tests and estimation of pyridine and permanganate values. The relationship of the results of these tests to the cystine content and swelling properties of the carrotted furs are given. B.K.

Application of Paper Chromatography to the Identification of Accelerators and Antioxidants. IV—Detection of Antioxidants. V—Detection of Accelerators and Antioxidants in Rubber of Unknown Composition

J. W. H. Zijp

Rec. Trav. chim., **75**, 1129-1136, 1155-1158 (Sept.-Oct. 1956)

Some antioxidants containing primary or secondary amino groups may be separated by descending paper chromatography after coupling with diazotised aminophenylsulphonic acids; phenyl- α - and phenyl- β -naphthylamine are discussed as examples. The method is of limited application. A wider range of such cpd. may be separated upon completely acetylated Whatman paper No. 1 using benzene-96% ethanol (1:1) as the mobile phase; the chromatogram is developed with a 4% soln. of benzoyl peroxide in benzene. The systematic and trade names, the developed colour of the spot, the R_f values, and the identification limits before and after chromatographic separation are tabulated for 14 antioxidants. These cpd. may be separated from a rubber sample by evaporation of the acetone extract, followed by re-solution in boiling 96% ethanol. Phenolic antioxidants may be separated upon the same acetylated paper. Two mobile phases may be used—butyl acetate—pyridine—methanol—water (1:5:1:3), and isopropanol—methanol—water (3:3:2)—and two colour developers—Millon's reagent and Tollens' reagent; by the application of all four it is possible to identify many phenolic antioxidants. The use of an ethanol extract of the rubber, and the addition of strontium chloride, prevent interference by stearic acid and paraffins. In the second paper (Part V) a detailed procedure is given, based upon the above findings, for the identification of accelerators and antioxidants in rubber mixes and vulcanisates of unknown composition. J.W.D.

Colorimetric Determination of Phenolic Resins

M. H. Swann and D. J. Weil

Anal. Chem., **28**, 1463-1465 (Sept. 1956)

The colorimetric determination of free phenols present in phenol-aldehyde resins in varnishes and other coatings is carried out by a modification of the nitrous acid method, using *n*-butyl acetate or 1-hexanol as solvent. The yellow colour obtained is specific for phenolic resins. Estimation of a resin is possible if its composition is known, and the phenol content of a resin of unknown composition may also be determined. L.T.W.

Acrylic Fibres (X p. 75)

XV— MISCELLANEOUS

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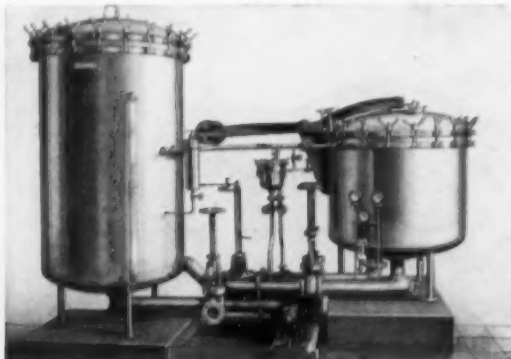
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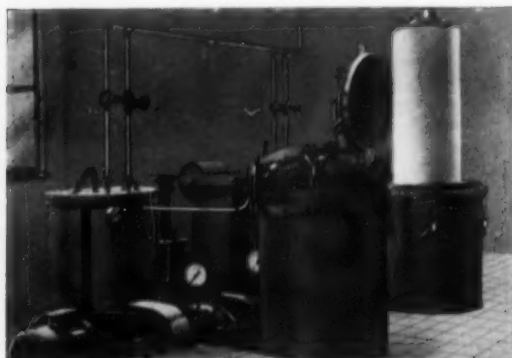
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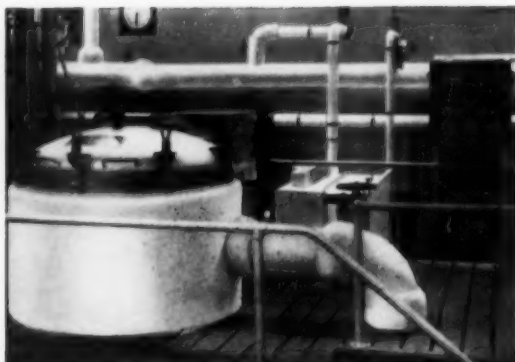
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Replies may be addressed Box —, THE SOCIETY OF DYERS AND COLOURISTS, DEAN HOUSE, 19 PICCADILLY, BRADFORD 1, YORKSHIRE, where all communications relating to these Advertisements are treated in strict confidence.

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THE MANCHESTER COLLEGE OF SCIENCE AND TECHNOLOGY

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Applicants should write in confidence to the Director of the Testing House at 10 Barlow Moor Road, Didsbury, Manchester 20.

THE MANCHESTER COLLEGE OF SCIENCE AND TECHNOLOGY

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MISCELLANEOUS

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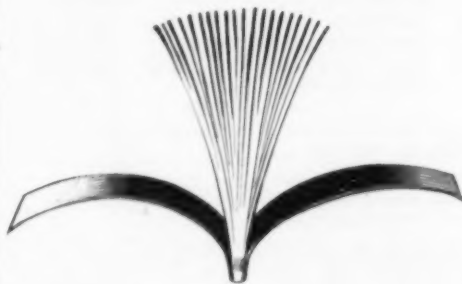
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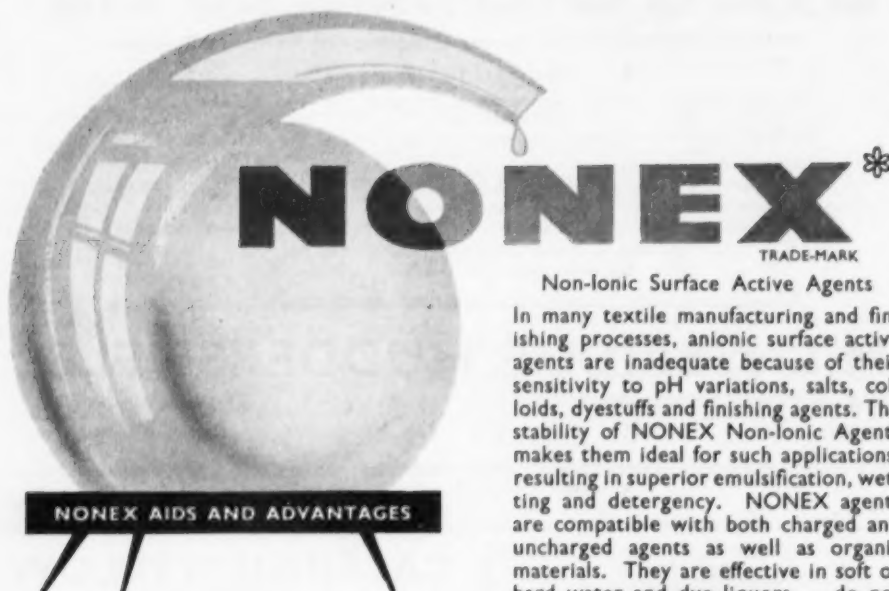
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For more information on NONEX write section 2-G

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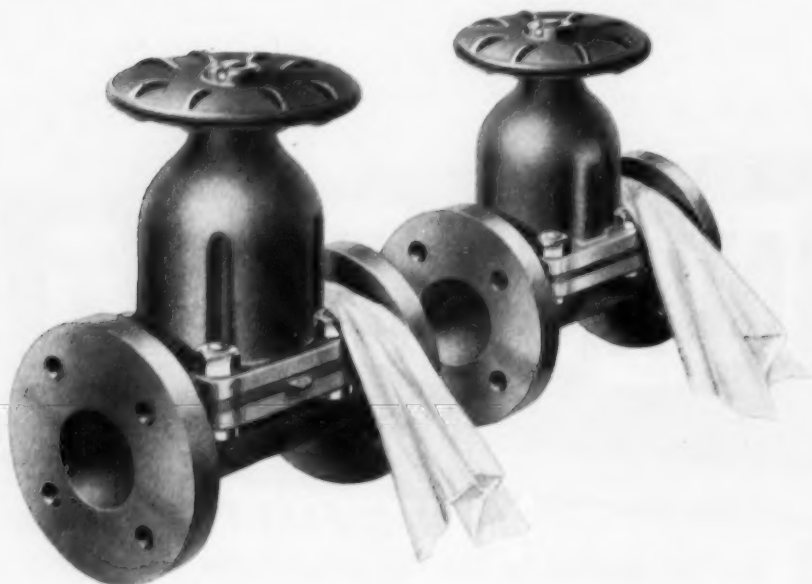
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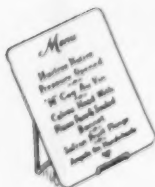
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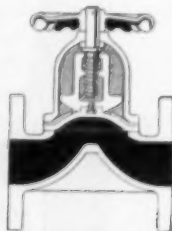
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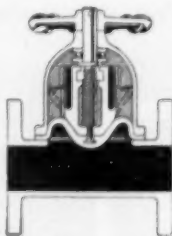
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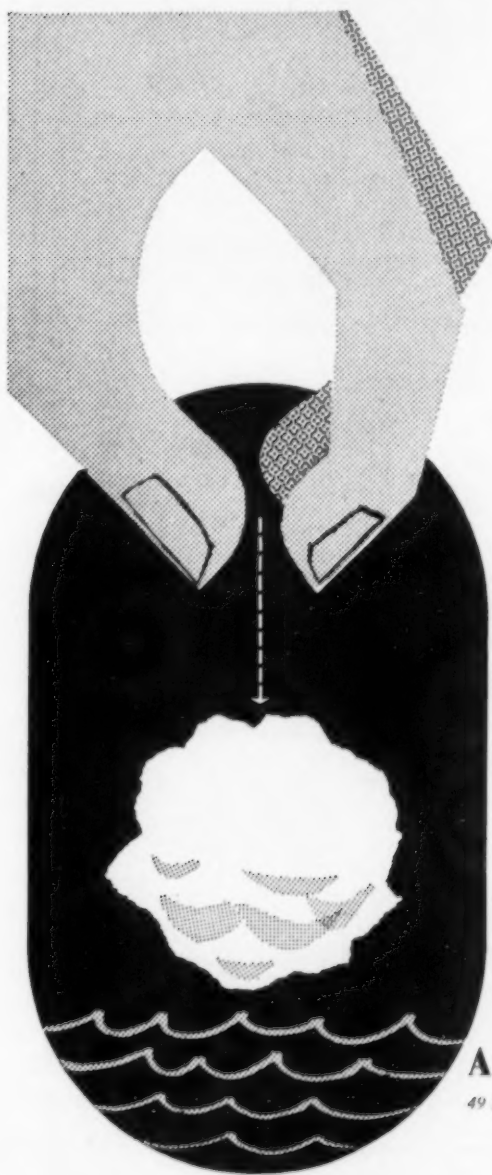
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For further information about Albright & Wilson's polyphosphates—Calgon* (glassy sodium metaphosphate), Polygon* (sodium tripolyphosphate) and Tetron* (tetrasodium pyrophosphate), write to:

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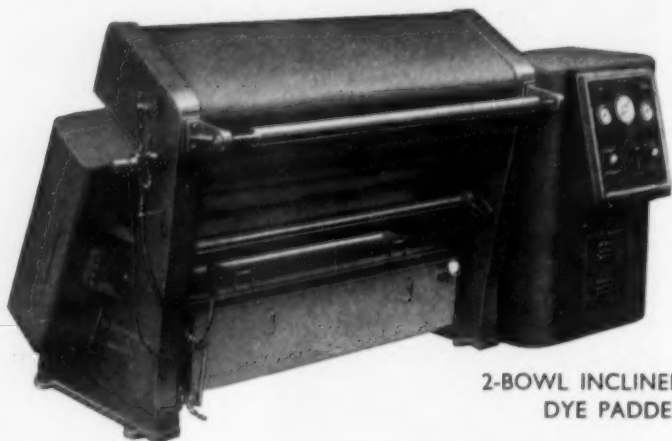
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